United States Court of Appeals

for the

District of Columbia Circuit

TRANSCRIPT OF RECORD

JOINT APPENDIX

166

IN THE

United States Court of Appeals

DISTRICT OF COLUMBIA.

No. 9576

THE LUBRI-ZOL CORPORATION, *Appellant,*

Lawrence C. Kingsland

~~CASPER W. OOMS~~, Commissioner of Patents, *Appellee.*

Appeal from the District Court of the United States for the District of Columbia.

United States Court of Appeals
for the District of Columbia
FILED SEP 1 1 1947
Joseph W. Stewart
CLERK

PRESS OF BYRON S. ADAMS, WASHINGTON, D. C.

INDEX TO JOINT APPENDIX

Page

I. Pleadings, Docket Entries and Other Papers.... 2
- Bill of Complaint to Issue Patent 2
- Answer to the Complaint 8
- Stipulation as to Plaintiff's Exhibit No. 2......... 10
- Informal Memorandum of Justice Letts 11
- Findings of Fact and Conclusions of Law 11
- Judgment 13

II. Excerpts from Testimony and Proceedings 14
- Opening Statement on Behalf of Plaintiff........ 14
- Opening Statement on Behalf of Defendant...... 16
- Witnesses:
  - Dr. Herman P. Lankelma 18
  - Dr. Kelvin Smith 32

III. Exhibits 38
- Excerpts from Plaintiff's Exhibit No. 1 38
  - Specification and Oath 38
  - Decision of the Board of Appeals............. 45
  - Examiner's rejection of all claims............ 47
  - Examiner's Statement 48
  - Affidavit of Albert K. Smith 54
  - Report on Affidavit 56
  - Examiner's Decision affirmed by Board of Appeals 59
  - Request for Reconsideration Denied 62
- Plaintiff's Exhibit No. 2: Chart A, Figures 1-10.. 65
- Plaintiff's Exhibit No. 3: Extracts from the file of the Downing et al. Patent No. 2,285,853.. 67
- Plaintiff's Exhibit No. 4: Chart B, Figures 11-12.. 71
- Defendant's Exhibit No. 1:
  - Downing et al. Patent No. 2,285,853............ 72
  - Malisoff Patent No. 2,289,509 77

IN THE

United States Court of Appeals

DISTRICT OF COLUMBIA.

No. 9576.

THE LUBRI-ZOL CORPORATION, *Appellant*,

v.

CASPER W. OOMS, Commissioner of Patents, *Appellee*.

Appeal from the District Court of the United States for the District of Columbia.

JOINT APPENDIX.

I.

PLEADINGS, DOCKET ENTRIES AND OTHER PAPERS.

United States District Court for the District of Columbia

Civil Action No. 30356

THE LUBRI-ZOL CORPORATION, *Plaintiff,* Box 3057, Euclid Station, Cleveland, Ohio

v.

CASPER W. OOMS, Commissioner of Patents, *Defendant.*

39 Filed Aug 27 1945

Bill of Complaint to Issue Patent

The Plaintiff, The Lubri-Zol Corporation, is a corporation organized and existing under and by virtue of the laws of the State of Ohio, with its principal office and place of business in the City of Wickliffe, State of Ohio.

I.

This Bill is filed in accordance with the provisions of the Federal Laws of the United States as provided for under Revised Statute 4915.

II.

The Defendant, Casper W. Oooms, is the Commissioner of Patents of the United States, a legal resident of the District of Columbia, and is sued in his official capacity as Commissioner of Patents of the United States.

40 III.

Heretofore, to-wit, on or prior to March 23, 1938, Carl F. Prutton, then a citizen of the United States and a resident of Cleveland Heights, State of Ohio, was the original, first and sole inventor of a certain new and useful improvement in Lubricating Composition; and said invention was a new and useful improvement not known or used by others in this country before his invention thereof, and not patented or described in any publication in this or any foreign country before his invention thereof or more than two years prior to his application for Letters Patent therefor, nor in public use or on sale in this country, for more than two years prior to his said application, nor ever abandoned; nor ever made the subject of any foreign patent application filed by him or his legal representatives prior to his said application; and said Carl F. Prutton, did on, to wit, March 23, 1938, duly file in the Patent Office of the United States an application in writing praying for the issuance to him of Letters Patent of the United States for said invention.

IV.

The said application has been passed upon by the Commissioner of Patents in the person of the Primary Examiner who allowed no claims and specifically refused to allow the following claims:

1. A lubricating composition comprising a mineral lubricating oil and a minor amount of an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

41 2. A lubricating composition comprising a mineral lubricating oil and a minor amount of an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms.

4. A lubricating composition comprising a mineral lubricating oil and a small percentage of an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

5. A lubricating composition comprising a mineral lubricating oil and a small percentage of an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms.

7. A lubricating composition comprising a mineral lubricating oil and from an effective amount to about 1% of an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

8. A lubricating composition comprising a mineral lubricating oil and an effective amount to about 1% of an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms.

10. A lubricant for metallic bearing surfaces comprising a major proportion of a mineral lubricating oil, and a minor amount of an alkylated aryl ester of an acid of phosphorus, in which the alkyl radicle contains at least four carbon atoms.

11. A lubricating composition comprising a mineral lubricating oil and a minor amount of a halogenated condensation product of an aromatic ester of a phosphorus acid with an unsaturated chain compound.

12. A lubricating composition comprising a mineral lubricating oil and a minor amount of tri-tertiary amyl phenyl phosphate.

42 13. A lubricant for metallic bearing surfaces comprising a major proportion of petroleum lubricating oil and a minor amount of an alkylated aryl phosphite having at least one alkyl group containing a plurality of carbon atoms.

14. A lubricant for metallic bearing surfaces comprising a major proportion of petroleum lubricating oil and a minor amount of an alkylated aryl phosphite having at least one alkyl group containing at least four carbon atoms.

15. A lubricant for metallic bearing surfaces comprising a major proportion of petroleum lubricating oil and a minor amount of a halogenated alkylated aromatic phosphite.

V.

The grounds upon which the Primary Examiner rejected said claims were:

(1) That each and all of the claims fails to define invention over the patents to Downing et al No. 2,285,853, and Malisoff No. 2,289,509.

(2) That claims 11 and 15 are lacking invention over the said patent to Malisoff.

VI.

The applicant thereupon duly appealed from the decision of the Primary Examiner to the Board of Appeals who affirmed the rejection of the Primary Examiner by its decision dated March 2, 1945.

VII.

The grounds upon which the Board of Appeals affirmed the rejection of said claims were the same as the grounds on which the Primary Examiner had based his rejection.

43

VIII.

Thereupon applicant duly filed a petition for reconsideration to the Board of Appeals, said petition pointing out that the disclosure of the Downing patent was improperly interpreted and pointing out by structural formulas wherein such disclosure had been misinterpreted. However, the Board of Appeals refused such request for reconsideration and reaffirmed the rejection of the Examiner by decision dated April 4, 1945.

IX.

The Commissioner of Patents has refused to allow any of the claims on the grounds that they are anticipated by aforesaid prior art.

X.

The plaintiff disagrees with the foregoing contentions of the Commissioner of Patents and contends that the claims are patentable over said prior art.

XI.

No appeal has been taken from the decision of the Board of Appeals to the United States Court of Customs and Patent Appeals; and no such appeal is pending or has been decided, and this Bill is filed within six months of the date of the refusal by the Board of Appeals aforesaid to allow the said application of the said Carl F. Prutton, Serial No. 197,732.

44

XII.

The aforesaid application of Carl F. Prutton, Serial No. 197,732, the invention covered thereby, and the patent to issue thereon, was, by an instrument in writing, duly assigned and transferred to The Lubri-Zol Corporation, an Ohio corporation, plaintiff herein; said assignment was duly recorded in the United States Patent Office at Liber L-196, Page 556 of the Transfers of Patents, and plaintiff is now the sole and exclusive owner of the entire right, title and interest in and to said application and the invention and discovery disclosed and claimed therein. The assignment aforesaid or a certified copy thereof is ready in Court to be produced and profert thereof is hereby made.

XIII.

The Commissioner of Patents has refused and still refuses to grant Letters Patent upon the aforesaid application, Ser. No. 197,732, and particularly claims 1, 2, 4, 5, 7, 8, and 10 to 15 inclusive thereof to which plaintiff is lawfully entitled.

Wherefore plaintiff brings this Bill under and in accordance with the Statutes in such cases made and provided and respectfully prays:

(a) That the Court adjudge and decree that Carl F. Prutton is the first, true and original inventor of the improvements set forth in the claims in said application Serial No. 197,732.

(b) That the Court adjudge and decree that plaintiff,
The Lubri-Zol Corporation, is entitled according to law to
receive Letters Patent of the United States for the
45 aforesaid invention and discovery of the said Carl
F. Prutton, as specified and pointed out and distinctly
claimed in said application Serial No. 197,732, as
the assignee of said Carl F. Prutton.

(c) That the Court authorize and direct the Commissioner of Patents to issue Letters Patent upon said application Serial No. 197,732 containing the claims thereof and any other claims which this Court may adjudge lawful and proper.

(d) To grant to plaintiff such other and further relief as equity may require and which to the Court may seem meet and proper.

(e) To issue a subpoena ad respondum directed to Casper W. Ooms, Commissioner of Patents of the United States under the seal of this Honorable Court requiring him to appear and answer this Bill of Complaint and Abide the Further Order of this Court.

THE LUBRI-ZOL CORPORATION

By KENT H. SMITH
President

EDWIN R. HUTCHINSON
Solicitor for Plaintiff
Edwin R. Hutchinson
1154 Munsey Bldg.
Washington 4, D. C.—NA 4904

OSCAR C. LIMBACH
Of Counsel
Oscar C. Limbach
Leader Building
Cleveland 14, Ohio

46 STATE OF OHIO
County of Lake, ss:

Kent H. Smith, being duly sworn, deposes and says that he is the President of The Lubri-Zol Corporation, the corporation named in the within entitled action; that he has read the foregoing Bill of Complaint and knows the contents thereof, and that the same is true to his own knowledge.

KENT H. SMITH

Subscribed and sworn to before me this 10th day of August, 1945.

R. J. FERREE,
Notary Public

(Seal)

R. J. Ferree, Notary Public
Lake County, Ohio
My commission expires March 9, 1946

* * * * * * * * * *

47 Filed Oct 6 1945

Answer to the Complaint

To the Honorable the Justices of the District Court of the United States for the District of Columbia.

Defendant admits the allegations of the unnumbered paragraph at the beginning of the complaint.

I. He admits the allegations of paragraph I.

II. He admits that he is the Commissioner of Patents and that he is sued in his official capacity as such. He denies that he is a legal resident of the District of Columbia. He states that his official residence is in the District of Columbia.

III. He admits that on March 23, 1938, Carl F. Prutton, claiming to be the original, first and sole inventor of certain alleged new and useful improvements in Lubricating

Composition, filed in the Patent Office an application for
patent thereon and in said application made aver-
48 ments corresponding to the allegations of paragraph
III. He denies, for reasons hereinafter given, that
the claims of said application set forth a patentable invention and denies that said averments would justify the issuance of a patent on said application.

IV, V, VI, VII. He admits the allegations of paragraphs IV to VII, inclusive.

VIII. He admits that applicant duly filed a petition for reconsideration of the decision of the Board of Appeals affirming the rejection of claims 1, 2, 4, 5, 7, 8, 10, 11, 12, 13, 14 and 15 of said application. He denies that in said petition applicant pointed out any improper interpretation or any misinterpretation by the Board of Apeals of the Downing patent. He admits that the Board of Appeals in response to said request for reconsideration reaffirmed on April 4, 1945, the rejection of said claims.

IX. He admits the allegations of paragraph IX but states that plaintiff is not lawfully entitled to receive a patent containing any of the claims set out in paragraph IV as it is deemed that the said claims are unpatentable in view of the following prior patents and for the reasons given in the statement of the examiner in answer to the appeal and the decisions of the Board of Appeals, copies of which will be furnished at the trial:

Downing et al.,	2,285,853,	June 9, 1942,
Malisoff,	2,289,509,	July 14, 1942.

Profert of copies of these patents is hereby made.

X. He admits that plaintiff disagrees with the decision of the Board of Appeals and that plaintiff contends that the claims are patentable over the prior art, but he denies said
contentions of plaintiff.

49 XI. He states, in answer to the allegations of para-
graph XI, that on May 14, 1945, the applicant Carl
F. Prutton filed in the Patent Office a Notice and Reasons

of Appeal to the United States Court of Customs and Patent Appeals from the decision of the Board of Appeals. He admits that this Complaint was filed within six months of the date of the refusal by the Board of Appeals to allow the aforesaid claims of plaintiff's application.

XII. He admits the allegations of paragraph XII.

XIII. He admits that he has refused and still refuses to grant letters patent upon said application including any one of the claims set out in paragraph IV. He denies, for reasons aforesaid, that plaintiff is lawfully entitled to a patent containing any of said claims.

W. W. Cochran
Solicitor, U. S. Patent Office,
Attorney for Defendant.

October 4, 1945.

* * * * * * * * * *

78 Filed Nov 26 1946

Stipulation

Whereas, the reporter's transcript of proceedings does not indicate that Plaintiff's Exhibit 2 was ever received in evidence as an exhibit in the above identified action; and

Whereas, counsel for the Plaintiff intended that it be so received in evidence and was under the impression that it had been offered and received; and

Whereas, counsel for the Plaintiff at the conclusion of the trial had asked the clerk whether all of the exhibits had been received and she on checking her records had advised that they had been received:

Therefore, it is hereby stipulated and agreed by and between counsel for the respective parties herein, this Honorable Court consenting, that Plaintiff's Exhibit 2 (photostat of chart) be considered as having been duly received in evidence at the time of trial of this action, and that it be accorded the same force and effect that it would have been accorded had it been duly offered and received at the time of said trial.

Edwin R. Hutchinson
Attorney for Plaintiff.

Nov. 26, 1946.

W. W. Cochran
Attorney for Defendant.

Nov. 26, 1946.

* * * * * * * * * *

79 Filed Dec 19 1946

Informal Memorandum

I find the claims in suit unpatentable over Downing and Malisoff.

Counsel for defendant will present an order dismissing the complaint.

F. Dickinson Letts
F. Dickinson Letts
Justice

* * * * * * * * * *

80 Filed Jan 31 1947

Findings of Fact

1. This is an action under R. S. 4915 (U. S. C., title 35, sec. 63) in which the plaintiff sought to have the Court authorize the allowance of claims 1, 2, 4, 5, 7, 8, and 10 to 15, inclusive, of his application No. 197,732 for patent on a lubricating composition.

2. The application here involved discloses a lubricant comprising mineral oil and a small amount of alkylated aromatic ester of an acid of phosphorus. Among the esters suggested are tri-tertiary amyl phenyl phosphate and tributyl phenyl phosphate.

3. The patent to Downing et al 2,285,853 discloses a lubricant comprising a mineral oil and a small amount of alkylated aromatic ester of an acid of phosphorus. Among the

esters suggested are tricresyl phosphate and di-lorol cresyl phosphate.

81 4. The patent to Malisoff No. 2,289,509 discloses a lubricant comprising a mineral oil and a small amount of halogenated tri-cresyl phosphate.

5. The diloral cresyl phosphate is an alkylated ester of a phosphorus acid of the type obtained by condensing a halogenated ester of a phosphorus acid with an unsaturated chain compound.

6. Diloral cresyl phosphate is an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms.

7. In view of the Malisoff patent no invention would be involved in halogenating any of the compounds disclosed by Downing.

8. There is no patentable distinction between the phosphite compounds set forth in the claims here involved and the corresponding phosphate compounds.

9. All the claims here involved are unpatentable in view of Downing's disclosure of the use of diloral cresyl phosphate as an added ingredient for lubricating compounds.

10. In view of the disclosure by both Downing and Malisoff of tri-cresyl phosphate as an added ingredient for lubricating compoistions, there would be no invention in using homologs of this compound such as tri-butyl phenyl phosphate for the same purpose.

11. None of the claims here involved defines in-
82 vention over the disclosure by Downing and Malisoff
of the tri-cresyl phosphate.

12. All the claims here involved are unpatentable over either the Downing or the Malisoff patent.

Conclusions of Law

1. Plaintiff is not entitled to a patent containing any of the claims set forth in the complaint.

2. The complaint should be dismissed as to all the claims involved.

F Dickinson Letts
Justice.

January 31, 1947.

* * * * * * * * * *

83 Filed Jan 31 1947

Judgment

This action came on to be heard at the last term and thereupon, upon consideration thereof, it is, this 31st day of Jan., 1947,

Adjudged that the complaint in this case be, and the same hereby is dismissed with costs against the plaintiff.

F Dickinson Letts
Justice.

Approved as to Form:
Edwin R. Hutchinson
Attorney for Plaintiff.

* * * * * * * * * *

II.

EXCERPTS FROM TESTIMONY AND PROCEEDINGS.

2 Opening Statement on Behalf of Plaintiff.

Mr. Limbach: This is a proceeding under R. S. 4915, following the filing of an application in the Patent Office on an application in which the claims were denied by the Patent Office.

The subject matter is a lubricant, a lubricant to which certain phosphorus compounds are added.

The application went to the Board of Appeals. The case was argued. The Board of Appeals gave its decision affirming the Primary Examiner. Then we petitioned for a reconsideration, and as a result of the reconsideration the Board of Appeals adhered to their decision, and I believe by reason of that second hearing before the Board of Appeals the issues have been clearly defined, and are really only two in number.

There are two patents on which the claims are rejected, one, a principal patent to Downing, and a secondary 3 patent to Malisoff. The claims here on file are divided into three groups. In order that you may have an indication as to the precise issue, if you will turn to the bottom of page 2 of the complaint, your Honor, the first claim is here set forth as:

"A lubricating composition comprising a mineral lubricating oil and a minor amount of an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound."

That type of claim you will note, your Honor, identifies the compound used in the oil by the mode of its manufacture, representing one of the types of claims here under consideration.

The other type of claim is represented, for example, by claim 2 at the top of the next page, which identifies an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms, merely by its chemical structure without referring to its mode of manufacture.

Then there are other claims as, for example, claim 11 which, in line 3, your Honor, you will note that refers to a halogenated condensation product. Those claims are similar to the claims of groups 1 and 2, with the exception that the compound also contains a halogen.

In the decision by the Board of Appeals on recon-
4 sideration wherein the two issues before us were
really affirmed, the Board of Appeals held with the applicant that certain disclosures with regard to mixed compounds, or mixed compositions were not anticipatory of the invention here claimed, but they did say that the applicant, and I am now referring to the opinion of the Board of Appeals, that the compound dilorol cresyl phosphate was in the opinion of the Board of Appeals an example of the type of compound that was covered by the claims under consideration and, therefore, each of the claims anticipated.

We are prepared to show by expert testimony that the Board of Appeals' interpretation of that disclosure was in error; that dilorol cresyl phosphate is not one of the compounds we are claiming and, therefore, it is not an anticipation of the claims.

The second issue is concerned with an affidavit filed by Dr. Smith, whom I have here and expect to call as a witness, in which he compared the effectiveness of the compound disclosed by the principal reference with the compounds, a representative compound covered by the claims here on file, and the Board of Appeals criticized the affidavit as being deficient in certain particulars. I say we believe the Board of Appeals did not understand fully the principal information given in the affidavit, and I propose to prove by calling Dr. Smith that the things that the Board of Appeals said should have been included in the
affidavit were actually there, and did show that the
5 compounds claimed were separate.

The Court: Mr. Reynolds?

Opening Statement on Behalf of Defendant.

Mr. Reynolds: May it please the Court, there are two references involved here which are quite similar. I think it will be enough to refer to the Downing patent at this time. That is the first patent in our folder. That shows the addition of certain materials to lubricating oil for the purpose of making it suitable for high pressure lubrication, and the only question here is whether the particular materials claimed are patentably different from the particular materials shown in the Downing patent. Downing lists a good many of them. There are two we rely on particularly. One is tricresyl phosphate, referring to page 2 of Downing in example number 3, reference to 5 per cent of tricresyl phosphate. That is one of the substances we rely on.

The other is referred to on page 3 of the patent in the first column. It is dilorol cresyl phosphate. Now, as regards dilorol cresyl phosphate, that is a compound of the exact type, I think, that is claimed here, with one exception, that is that it has only one carbon atom in the alkyl group, whereas the claims here, one way or another, require that there be two alkyl atoms. Our claim is that that is an obvious addition which does not involve invention because
it is common in this art to refer to what is known
6 as two homologous series which differ from each
other only by addition of CH_2 or multiples of CH_2;
that is, we start with one carbon atom, and the corresponding CH_2, and 3, and so forth, and that is recognized generally in the art, so that we feel that mere suggestion by Downing that one member of this group, that is, a member having one carbon atom, would be suitable for this purpose, and suggests that the others, 1, 2, 3, 4, and so forth, would also be suitable for that purpose according to the application filed, and he also suggests that compounds that have only one carbon atom are suitable, so that the application itself recognizes that there was a relationship between them and, therefore, we feel that the disclosure of

tricresyl phosphate is such as to render these claims unpatentable, he has merely discovered that it is suitable.

The other, diphenyl phosphate, we contend it is exactly the kind considered here, that the claims presented here read directly on that. I imagine that will be gone into in the testimony and I won't undertake to comment on it now.

As Mr. Limbach stated, this compound is halogenated. That is in his claim, and that is common, halogenation, as is shown in Malisoff, so I think clearly that they are not patentable at all.

Mr. Limbach: Your Honor, I would like to call as my first witness, Dr. Herman Lankelma.

We have made a chart, your Honor, which we
7 tacked on the board for use by the witness in order
possibly to facilitate the testimony, and we have also a pin-up board which the witness may use to construct some of these compounds, and I have also here a box of tags which we hope to pin on the board to illustrate what these compounds are like.

Now, I have here a photostat, which is a picture, as you see, of the board on the right-hand side, namely, the one that has already been made up.

Prior to the introduction of that as an exhibit I should first like to introduce a certified copy of the file history of the application involved as plaintiff's exhibit number 1.

The Court: It may be marked and received.

(Thereupon, document entitled File Wrapper and Contents in the matter of pending application of Carl F. Prutton, filed March 23, 1938, serial number 197,732, was by the Court received in evidence and marked plaintiff's exhibit number 1.)

Mr. Limbach: I would like to offer in evidence as plaintiff's exhibit 2 the photostat of the chart, or I will withhold offering that until after the witness has referred to it. That is the one I gave you a moment ago.

Now, I will hand the witness one of these also because on the photostat of the chart on the board I have applied figure numbers for facility and identification of them in the record. Thereupon—

8 **Dr. Herman P. Lankelma** was called as a witness for and on behalf of the plaintiff, and being first duly sworn, was examined and testified as follows:

Direct Examination

By Mr. Limbach:

Q. Now, Dr. Lankelma, will you please state your full name, age, residence, and occupation? A. Herman P. Lankelma, age 50; I live at 22575, Calverton Road, Shaker Heights, Ohio. I am professor of chemistry at Western Reserve University in Cleveland.

Q. And what were your academic training and other attainments, Dr. Lankelma? A. I received a Bachelor of Art Degree in 1918 from Central College, Pella, Iowa, and majored in chemistry at the State University of Iowa, and received the Master of Science Degree in Chemistry in 1921 and continued my studies and received the degree of Doctor of Philosophy in Chemistry at the State University of Iowa Iowa City, Iowa in 1923.

Q. How long have you been in your present position, Dr. Lankelma? A. Since 1923.

Q. Do you have any substantial amount of work in organic chemistry? A. Yes, my work is confined en-
9 tirely to teaching and research work in organic chemistry.

Q. You heard me a moment ago read the counts or the claims which comprise the issue here on appeal. One of those identifies the addition agent as an alkylated aromatic ester of an acid of phosphorus of a certain type? A. Yes.

Q. Now, by having reference to the chart on the board can you explain to the Court what we mean by an ester of an acid of phosphorus? A. An ester of an acid of phosphorus would have one, two or three of the hydrogen atoms of the acid replaced by a hydro-carbon group.

Mr. Limbach: The witness referred to figure 1 on the chart.

By Mr. Limbach:

Q. You may explain, if you will, please, the various things we have put on this chart before you go further with your explanation. A. The esters of the acids of phosphorus consist of two basic units, and these units are, first, the acids of phosphorus which are shown here, and second, hydrocarbon groups; the hydrocarbon groups pertinent to the esters under consideration are also shown here.

Now, first, we have the two acids of phosphorus. They are known as phosphoric acid and phosphorus acid. They differ in oxygen content. Phophoric acid contains one atom of oxygen more than phosphorous acid.

Now, these acid hydrogen atoms are replaceable by other groups; there are three acid hydrogens, each of which is replaceable by an organic group.

The phosphoric acid without the hydrogen atoms, which atoms are replaceable, is known as the phosphate group.

We have the same situation in phosphorous acid in which hydrogen may similarly be replaced, and that group with one, two, or three of its hydrogen atoms replaced is known as phosphite group.

Now, the organic groups which may replace the hydrogen atoms of phosphoric and phosphorous acids to give the phosphate and phosphite esters are shown in these figures.

The first is the hydrocarbon, or aromatic hydrocarbon benzene. It is liquid and resembled ordinary gasoline very much. The hydrogen atoms of benzene are replaceable with these groups (indicating).

Now, the removal of one hydrogen atom and its replacement by another group, such as the phosphate or phosphite group, leaves us with the organic phenyl group. This next row shows hydrocarbons, which in contrast to benzene are acyclic in structure. The molecule is without ring structure. The hydrogen atoms of these hydrocarbons, like those of benzene, are replaceable leaving hydrocarbon groups. The first of these hydrocarbons is methane; it

comprises the large part of natural gas which is used as
a fuel, somewhere in the neighborhood of 90 per cent
11 or more of natural gas is composed of this hydro-
carbon, and if one of its hydrogen atoms is replaced
the remaining group is known as methyl.

In the next figure we have a hydrocarbon chain called butane. It is also a gas, and it constitutes a type of gas you have in bottled gas, for example.

Now, in butane, the hydrogen atoms are also replaceable, the same as in methane, leaving the butyl group which is shown here (indicating).

This hydrocarbon represents an unsaturated compound known as butylene related to butane in that it contains two less hydrogen atoms. These hydrogen atoms are removed from adjacent carbon atoms, the valence bond of which combine to give the double bond shown in the figure. This double bond is a point of unsaturation in the molecule.

The addition of one hydrogen atom at this point of unsaturation or the removal of one hydrogen atom from the hydrocarbon butane lead to the same group, the butyl group shown in figure 9.

Now finally, as shown here, we have the lorol group, which is mentioned in the Downing patent, and it is shown here as a chain of twelve carbon atoms. That group may be defined as one containing approximately eight, ten or twelve carbon atoms; it is a mixture of alcohols obtained by reduction of the acids of cocoanut oil, or related oils.

Now, the finished molecule, the esters of the phosphorus
acids, simply consist of combinations of these units which
I have shown. We may show a combination of acid
12 units with these hydrocarbon groups and they may
be illustrated by these pin-ups which are in the form
of exhibits.

We can start first with the phosphate group, from phosphoric acid. This is the group which we say has three replaceable atoms identical with the group shown here on the chart.

Now, they may be combined, for example, with the phenyl groups this way, and this is readily done as a chemical process, and you obtain the finished ester, in this case triphenyl phosphite, three phenyl groups combined with a phosphate group.

Now, the hydrogen atoms—

Mr. Limbach: Pardon me. Do you have any question thus far, your Honor?

The Court: No, not now.

The Witness: The hydrogen atoms of this hydrocarbon group, as we saw before, are replaceable, for example, we can replace a hydrogen atom of this phenyl group by a methyl residue and then we have a mixed ester of phosphoric acid, di-phenylcresyl phosphate.

In this case a combination of phenyl and methyl is called a cresyl group. That illustrates the mixed ester in which the groups which are replacing the hydrogen of phosphoric acid are not the same and we therefore have a mixed ester.

By Mr. Limbach:

Q. I beg pardon. What would that be called? A.
13 Called diphenylcresyl phosphate. To illustrate tri-
cresyl phosphate, it would simply have a hydrogen atom of all three of the phenyl groups replaced by methyl groups. Each of these now is a cresyl group, and that would illustrate tricresyl phosphate. That is no longer a mixed ester; the organic groups are all cresyl groups.

Now, if instead of one of these cresyl groups we have a lorol group we now have a mixed ester, that is, we have lorol dicresyl phosphate.

If instead of a second cresyl group we have a second lorol group we now have dilorol cresyl phosphate, which is the phosphate ester, figure 11 of the chart, a mixed ester with 2 aliphatic groups, and an aromatic group substituting for the hydrogens of the phosphate group.

Mr. Limbach: At this point, your Honor, I would like to interrupt the witness. The Board of Appeals in its decision upon reconsideration said that in their opinion dilorol cresyl phosphate was a compound in which these lorol groups were attached to the cresyl group.

By Mr. Limbach:

Q. Would you construct a compound such as the Board of Appeals had in mind, Doctor, please? We don't have enough of these? A. We don't have enough of these. I can construct one such group.

Q. Construct what the Board of Appeals presum-
14 ably had in mind. A. They presumably had in mind
these cresyl groups would have hydrogen atoms of
the cresyl group replaced by the lorol groups.

Mr. Limbach: In other words, if your Honor will look at the decision of the Board of Appeals on reconsideration, that is one of the principal points here. It is in evidence. Mr. Reynolds will probably give it to you. That is the decision of April 4, 1945.

The Court: Yes, I have it here.

Mr. Limbach: On the first page, in the first paragraph in line 5, "The same is not true of the dilorol cresyl phosphate. This should mean a compound where the two lorol radicals of fifteen carbon chains each are attached to the benzene ring of the cresyl radical rather than to the phosphoric acid radical."

As you see, on this chart is attached the benzene ring, that is this ring (indicating) which is derived from benzene, and there they are attached to the cresyl radical. You see, this is the cresyl radical that is on a phosphate group. In other words, the Board says they are attached down here rather than being attached up here (indicating) and that is really the issue that we are talking about so far as this compound is concerned.

Now, in an endeavor to find out what the Patent Office had in mind, I looked at the file history of the Downing
patent, and I offer in evidence a certified copy of
15 excerpts of the file history of the Downing patent.

Mr. Reynolds: I object to that. It is incomplete. I think the whole record should be put in if parts are to be relied on.

The Court: I suppose the whole record will be introduced?

Mr. Reynolds: It is available.

Mr. Limbach: I think, your Honor, that the whole record of the Downing patent, everything but what we have here, would contain considerable material which would be entirely redundant to this issue. The single point we are concerned with here is covered, as I shall show, by the excerpt here which I offer in evidence and ask to have marked plaintiff's exhibit 3.

Mr. Reynolds: Of course, I do not want to be technical, but there may be other parts in there that will tend to contradict this, but I am willing that this be admitted subject to our right to introduce other parts later.

Mr. Limbach: That will be satisfactory.

The Court: It will be received.

(Thereupon, extracts from the Downing Patent, No. 2,285,853 was by the Court received in evidence and marked plaintiff's exhibit number 3.)

Mr. Limbach: This is important for the following reason; on the first page you will find a claim 61 which, when the patent issued, was renumbered as claim 2, and the witness also has a copy of this, so I ask him to follow that,
and this is over on the second page, claims 80, 81
16 and 82, which were renumbered in the patent as
claims 12, 13 and 14.

Now, claim 61 identifies the addition agent is a minor amount of a mixed ester of an acid of phosphorus, which ester contains at least one radical of a phenol and at least one radical of an acyclic alcohol.

Then over on the next page, claim 82 identifies the addition agent as dilorol cresyl phosphate, which is the compound we have here under consideration.

Then on the last page of this abstract, the third line from the bottom, it says claim 61—now, this is the attorney for Downing explaining to the Patent Office what the compounds were and what the claims meant—"Claim 61 is generic to each of claims 79 to 82."

In other words, the attorney for Downing said that claim 82, which identifies the addition agent as dilorol cresyl phosphate, was a species of claim 61 which identifies the ester as a mixed ester of an acid of phosphorus, and so forth.

By Mr. Limbach:

Q. Now, I would like to ask the witness whether the Board's interpretation of dilorol cresyl phosphate with both lorol groups attached to the benzene ring of the cresyl group is a mixed ester? A. It is not.

17 Q. Is it a mixed ester which contains at least one radical of phenyl, and at least one radical of an acyclic alcohol? A. It is not.

Q. Can you elaborate on your answer, Dr. Lankelma? A. Well, this ester as it stands would be—it is not a mixed ester because we do not have a different group replacing the hydrogen atoms of phosphoric acid, and this ester as it stands would not be called dilorol cresyl phosphate. That is if it were named a dilorol cresyl phosphate it would not be properly named. To be properly named, as this structure is here, it would have to be called methyl-dilorolphenyl phosphate.

Q. What would be the structure of it if it were a mixed ester? A. If it were a mixed ester these lorol groups, the lorol alcohol, would be combined with the phosphate group in that way, and that would constitute a mixed ester in which we have two different groups replacing the hydrogen of the phosphoric acid, in one case a lorol group and in the other case a cresyl.

Q. In other words, that is a dilorol cresyl phosphate? A. A dilorol cresyl phosphate.

Q. I think we have a tag for that.

Now, of the compounds that the applicant discloses
18 as an example of his alkylated aromatic esters of an
acid of phosphorous is tributyl phenyl phosphate.
Will you construct that for the Court, please? A. That structure is based on the phosphate group which calls for a substitution of three hydrogen atoms. The finished molecule as it stands here is triphenyl phosphate.

Now, tributylphenyl phosphate would have one of each of the hydrogen atoms of the penyl groups replaced by a butyl group. We have a butylphenyl group, the same group, butylphenyl—butylphenyl (indicating); butylphenyl, therefore, tributylphenyl phosphate (indicating).

Mr. Limbach: For the convenience of the Court I have a photograph which shows the chart as just completed by the witness which I shall ask that it be marked as plaintiff's exhibit 4.

The Court: Without objection it may be received.

(Thereupon the chart referred to was by the Court received in evidence and marked plaintiff's exhibit number 4.)

By Mr. Limbach:

Q. Now, is triphenyl phostphate an aromatic ester of phosphorus? A. It is.

Q. Now, will you illustrate for us and show how triphenyl phosphate is an aromatic ester of phosphorus? A. The
three phenyl group constitute the aromatic portion,
19 an aromatic nucleus of an aromatic hydrocarbon.
These constitute the butyl groups, which are derived from butane and butylene, if for example the triphenyl phosphate were akylated with butylene, which would add to the phenyl groups the common butyl group, the result would be tributylphenyl phosphate an alkylated aromatic ester of phosphoric acid.

Q. In your description just given you referred to the use of butylene in the alkylation process for the production of tributylphenyl phosphate.

Is butylene an unsaturated compound? A. Yes, it is an unsaturated hydrocarbon.

Q. Is that process you have just described then like the wording of claim 1 which identifies the ester as that type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound? Would you want to repeat that? A. Yes, it would be an aromatic ester of phosphoric acid condensed with an unsaturated chain compound.

Q. Is it possible to make a tricresyl phosphate by condensing an aromatic ester with an unsaturated chain compound? A. It is not, because in order to have an unsaturated chain you must have two or more carbon atoms. The acyclic carbon atom here would not constitute an unsaturated chain. Your minimum is two carbon atoms
20 whereas methyl which is found in the cresyl group
contains only one.

Q. In order to be sure that we have of record the principal definitions of some of the terms, will you indicate again for the Court the structure of the phosphoric acid? A. Phosphoric acid is the structure which is shown here (indicating).

Q. Is that figure 1 of exhibit 2? A. Yes, that is figure 1.

Q. And where is the phosphorous acid? A. That is shown in figure 2 of exhibit 2.

Q. And which is the term which generically defines both of those types of acid? A. Acids of phosphorus.

Q. And what is an aliphatic hydrocarbon? A. An aliphatic hydrocarbon is a hydrocarbon in which the carbon atoms are not in the form of a ring; to state it positively the carbon atoms are in the form of a chain, or joined to form a chain.

Q. Is that synonymous with acyclic? A. Yes.

Q. And what is an aromatic hydrocarbon? A. An aromatic hydrocarbon is one which is based on this ring structure of benzene.

Q. When we say aryl hydrocarbon what do we mean? A. It is a broad term, based upon the aromatic hydro-
21 carbon benzene.

Q. What is a phosphate? A. A phosphate is a product which is obtained by replacing one, two or three of the hydrogen atoms of phosphoric acid by some group; if it is a hydrocarbon group the product is an organic phosphate.

Q. What is phosphite? A. Phosphite is the same type of compound based on phosphorous acid.

Q. Now, just to be sure that the Court understands, I would like to ask you to define again the difference between a mixed ester and one which is not a mixed ester. A. In a mixed ester of a phosphorus acid at least two of the groups replacing the hydrogen atoms of the acid are different. In an ester which is not mixed the groups are the same; there is no difference in the groups replacing the hydrogen.

Q. One further question; in a homologous series where compounds differ from one another, what can you say, for example, as to the increase or decrease of one carbon atom per step; where do you usually find the greatest difference between adjacent members of a homologous series? A. Between the first member and the second, that is, between the single carbon member and the following member which contains two carbons.

22 Q. Have you any illustration which bears out that statement? A. Yes, various possible illustrations. The two alcohols, methyl alcohol and ethyl alcohol could be used. For illustration, methyl alcohol is commonly known as a wood alcohol. It is a poison and carries a poison label. Ethyl alcohol is ordinary grain alcohol and is used for beverage purposes.

Mr. Limbach: You may cross examine, Mr. Reynolds.

Cross Examination

By Mr. Reynolds:

Q. Now, referring to that legend dilorol cresyl phosphate which you have set up there, you have shown the lorol as attached to the oxygen rather than to the cresyl, is that right? A. Yes, sir.

Q. Now, in tributylphenyl phosphate, then, you would say the tributyl is attached to the phenyl, isn't that right, rather than to the oxygen? A. Yes.

Q. Why is it that in this and similar analogies you consider a different form of attachment is indicated? A. Because the phenyl is a basic unit, and if you put butyl groups on it you say butyl phenyl. If you put a methyl group on it you call it cresyl, but if you substitute further hydrogen atoms you go back to your basic unit and it becomes
23 a methyl dilorophenyl phosphate.

Furthermore, if the lorol groups were on the phenyl and you had that single substituted group you would have an acid phosphate, you would have to call it a mono dilorolcresyl phosphate, or you would have to call it a dilorolcresyl acid phosphate because there are still acid hydrogen atoms remaining on the phosphate group.

Q. Now, referring to that compound again, which you say is dilorol cresyl phosphate, is that an alkylated aromatic ester of phosphorous acid, or not? A. This is an alkyl group, (pointing to the methyl group) and that is an aromatic group (indicating the phenyl group) and to that extent is an alkylated aromatic ester.

Q. Does it contain an alkyl group containing at least two carbon atoms? A. It contains an alkyl group having more than two carbon atoms.

Q. Then that satisfies this statement, doesn't it, an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms? A. An alkylated aromatic ester of phosphoric acid. In that case it would be an alkylated aromatic ester, acid ester of phosphoric acid.

Q. Well, an acid ester is an ester, isn't it?

The Court: What is the question?

Mr. Reynolds: I asked him if an acid ester wasn't
24 an ester.

The Court: Oh yes.

The Witness: Well, it is an ester and it is an acid.

The Court: It is an ester what?

The Witness: And it is an acid?

By Mr. Reynolds:

Q. Well, you haven't answered the question categorically, that is, an ester of the type I have just described, would you say it is or isn't? A. Well, it is an ester, and it contains an alkylated aromatic group.

Q. And it, as an alkyl group, contains at least two carbon atoms? A. Yes.

Q. Now, you have been asked about claim 1 which contains a reference to an alkylated aromatic ester of an acid of phosphorous of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

What do you understand that type to mean there? A. That type of an aromatic ester would be one which had the phenyl group substituted for hydrogen of phosphoric acid, that is the phenyl group replacing the hydrogen of phosphoric acid.

Q. And is tricresyl phosphate, isn't it? A. Tricresyl phosphate as an aryl group, the aromatic groups replacing the hydrogen or phosphoric acid.

Q. Well, would you say that tricresyl phosphate is an ester of a type obtained by condensing an aromatic ester of a phosphorous acid with an unsaturated chain compound? A. I would not. You do not have an unsaturated chain which you could use to give you a methyl group.

Q. Now, do you consider that that language means that it was actually formed in this way, or that some authorities form it in this way? A. Well, you form it this way in actual practice; whether you considered formed in that way theoretically, it doesn't—

Q. Well, now, would you consider that tricresyl phosphate satisfies that requirement: A. What requirement, the unsaturated hydrocarbon condensed with the aromatic?

Q. Yes. A. No.

Q. It does not? A. No.

Q. I am not quite clear whether you think that it is necessary that it should be formed in that way, or only that it is similar to some others that can be formed in that way.

Mr. Limbach: I don't quite understand the point, Mr. Reynolds, of the question.

26 Mr. Reynolds: I will start over.

By Mr. Reynolds:

Q. You said an ester obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound, that would mean that it was actually formed in that way, wouldn't it? A. Yes.

Q. Now, when you say that type obtained in that way, it is a broader statement, isn't it? It means similar to others that are formed in that way, doesn't it? A. I don't see why it should.

Q. What do you mean, what do you think that type means? A. Just what it says.

Q. You think it means exactly the same thing as if that type were omitted? A. Yes.

Q. Then you think that type just means nothing in that connection, is that right? A. Well, it leads to a type of ester.

Q. Well, now, for instance in the application here there is reference to a condensing agent of the type just illustrated by anhydrous aluminum chloride.

Do you think that limits it to anhydrous aluminum chloride? A. I would say that in this case it is based on the
action of the agent, that is, any agent which will
27 bring about the condensation of the hydrocarbon
with an aromatic nucleus. Maybe you will find various interpretations.

Q. That is what I am trying to get, your interpretation of it in this connection. A. I would say it would be a condensing agent having catalytic action like that of aluminum chloride; a chemist would interpret that, I would, as agents which had that type of action.

Q. That would exclude tricresyl phosphite, would it, as well as tricresyl phosphate? A. Oh, yes.

Q. Now, will you set up the tricresyl phosphate again, please? A. Yes.

Q. Now, that could also be called trimethyl phenyl phosphate, is that right? A. Yes, sir.

Q. And how is that related to tri butylphenyl phosphate? A. The difference is in the length of the carbon chain of the substituted butyl.

Q. In other words, one is a homolog of the other? A. The butyl group is a homolog of the methyl.

Q. Would you expect them to have similar properties or dissimilar properties? A. It depends on what you mean by similar or dissimilar.

28 Q. Well, consider it the way you want. A. They might look alike but any chemist can distinguish them by their physical properties, the same as he can distinguish methane and butane. Methane and butane are very similar but any chemist would be able to distinguish them easily.

Q. Isn't it true that homologs are similar to each other? A. Yes, they bear similarities to each other.

Q. You don't think that that one part of the series has homologous properties and that the other doesn't? A. It depends entirely on the case.

Q. Of course, there are exceptions, but what I am asking you is about the change. A. There are exceptions in every case. It depends on the properties.

Q. Suppose you didn't have anything about the property, never had tried it before. Here we know part of the homologous property, you would be able to get the others, wouldn't you? A. I wouldn't say that.

Q. Wouldn't you say it was a similar property? A. I wouldn't say.

Q. I will read you a statement from Wertheim, textbook of organic chemistry, 1939, by the Blakiston Company of Philadelphia, with reference to homologous parts.
29 He says, "These compounds have similar chemical traits, because their structures are closely related;

therefore we can learn the chemistry of the entire group with no more effort than would otherwise be required to study a single compound. Of course we may anticipate certain exceptions to this rule but such exceptions will make very little trouble.''

Do you think that is a fair statement, or not? A. I don't think it is. That is not the way I would state it, sir.

Q. Now, coming back once more to the tricresyl, the application that is involved here contains this statement, "The following is a list of examples illustrating, but not intended to limit the type of addition agent contemplated by this invention,'' and then listed is tricresyl phosphate, and that is listed with tri butylphenyl phosphate and so on.

Now, will you say that tricresyl phosphate is the same type as tributyl phenyl phosphate, or not? A. They do have one feature in common, that is, they are both alkaryl esters of phosphoric acid.

Q. You would say that tricresyl phosphate is of the same type as tributyl phosphate, is that right? A. It depends on how you use the word "type".

Q. Well, take it where it is used in column 1. A. If you want to use the word "type'' you may have alkyl esters of phosphoric esters there.

Q. Would you say that tricresyl phosphate has the same relation to tributyl phenyl phosphate that tri butyl phosphite has to tributyl phenyl phosphite? A. Yes.

• • • • • • • • • •

Dr. Kelvin Smith, a witness called for and on behalf of the plaintiff, being first duly sworn, was examined and testified as follows:

Direct Examination

By Mr. Limbach:

Q. Will you please state your full name, address and occupation? A. Kelvin Smith.

Q. Your residence? A. 22099 Shaker Boulevard, Shaker Heights, Ohio, vice president in charge of research and engineering of the Lubri-Zol Corporation.

Mr. Limbach: Your Honor, this witness has filed an affidavit,
has made an affidavit of record in this case.
31 The Primary Examiner in his report to the Board
of Appeals on that affidavit considered that the affidavit
was in certain respects deficient.

The Board of Appeals in its reconsiderdation also held that it was in certain respects deficient in that it did not show certain factors they held it was necessary to prove, and this witness' testimony is now to show that the data which his affidavit originally had in it, when properly considered in the light of those skilled in the art, did have in it the data which the Board of Appeals was actually looking for.

When I gave Dr. Smith the affidavit to read this morning in order to refresh his recollection of the facts in the affidavit he pointed out that his original affidavit contained certain typographical errors, particularly in connection with a quotation made there from the application to which it refers.

I have had the body of the affidavit re-typed in order to correct those errors. Dr. Smith would like to have in the record in the affidavit a page which does not contain those typographical errors. I therefore ask that this paper be marked plaintiff's exhibit 5.

Mr. Reynolds: I do not understand just what this is offered for.

Mr. Limbach: That is merely that somewhere in the
record—there is no change in the substance whatsoever,
32 but Dr. Smith, being a meticulous and careful
man, hated to see his name on anything with errors
in it, and wanted it corrected. I don't care whether it goes
in or not.

The Court: I think you could have him testify as to any errors without having it received as an exhibit in this case.

Mr. Limbach: We will do that; we will do it that way.

The Court: I suggest that you avoid using any of your time that is unnecessary.

Mr. Limbach: I think under the circumstances, because we are pressed for time, we will waive that, your Honor.

The Court: We adjourn at 4 o'clock.

By Mr. Limbach:

Q. I hand you a copy, Dr. Smith, of the affidavit that is of record in the certified copy of the file and ask you to refer to the tabualted data which is attached thereto.

In the Examiner's report of that affidavit the Examiner complained that the claims here in issue cover those compounds in which the alkyl group here contains two or more carbon atoms, and that you selected for test one which contains four carbon atoms.

Will you explain why that selection was made? A. There were several reasons; one is that it could be prepared easily from commercially available materials. Another is that it was more properly representative of the entire class of compounds claimed than would be one with two carbon atoms in the alkyl group.

33 The third is that, as Dr. Lankelma pointed out, since the greatest change in the homologous series takes place between the first member, which has only one carbon atom, and the second which has two, there could be expected to be much less change between the member which has four carbon atoms and one which has two than between the group of those with two or more carbon atoms and the one which has only one carbon atom in the chain.

Q. In other words, the Examiner said that you used butyl with four carbon atoms. Why did you use that? A. That is a different question.

Q. What is the answer to that question? A. As to why butyl phenyl phosphate was used rather than amyl phenyl phosphate, it represents material which has a greater amount of carbon atoms in the chain than that described in claim 1 but the least amount of carbon atoms in the chain described in the most specific claim which says the alkyl group should have at least four carbon atoms.

Q. Now, on the two columns on the right-hand side of that data sheet it is indicated that .25 per cent of tri phenyl phosphate was used in comparison with .33 per cent of butyl phenyl phosphate with which it was compared, and the Examiner stated that for comparison equal amounts should have been used.

Why were different amounts used? A. In making comparisons between chemical compounds it is customary to make comparison on a molecular percentage basis,
34 for example, if there is a difference in the molecular
weight of the two compounds, what are called equivalent amounts would be used in making the comparison.

Now, tricresyl phosphate has an approximate molecular weight of 370, whereas tri butyl phenyl phosphate has an approximate molecular weight of 495, so that comparing those two compounds on an equivalent basis, (that is, with the same number of molecules per unit of volume, or unit of weight), if we are going to use .25 per cent of tri-cresyl phosphate we should use .25 multiplied by the ratio of 495 divided by 370 or .33 per cent of tri-butyl phenyl phosphate, and that is what we have done in this series of tests.

Q. The Board of Appeals on reconsideration in its decision stated that they could not find any of the data pointing to improvement as to any oxidation effect. Is it there? A. Yes, it is here in a number of places, for example, the Conradson carbon test. The Conradson carbon test consists of igniting oil in a standard type of equipment in an iron tube to obtain the carbon residue which is left in the tube after the sample has been burned. The residue is a measure of the deterioration of the oil and the amount of oxidation it has undergone and, therefore, constitutes the kind of information that I believe was referred to as the degree of oxidation.

Another one would be the naphtha insolubles and chloroform solubles.

35 For example, under naphtha insoluble the material determined is that which at the end of the oxi-

dation test is insoluble in a standard hydrocarbon solvent and, therefore, represents the sludge that has been generated, the sludge being the hydrocarbon insoluble materials formed by the oxidation test.

Q. The Board of Appeals also said that there was no showing of data there with respect to sludge prevention. Have you covered that sufficiently in your last answer? A. The two things are related to some extent and, as I have already said, the naphtha insolubles are definitely a matter of sludge resulting from oxidation.

There is one further thing that I omitted mentioning in the oxidation test and that is the acid number. The acid number definitely indicates the degree of oxidation since it represents one of the end-products of the oxidation. In other words, the higher the acidity of the oxidized sample the more indication of oxidation there is that has taken place during the oxidation process.

Mr. Limbach: You may cross examine.

Cross Examination

By Mr. Reynolds:

Q. Now, these tests indicate that tricresyl phosphate is useful as an addtiion agent for lubricating oil, do they not? A. Yes, sir.

Q. And your position is that they show the tri-butyl phenyl phosphate to be superior in some respects,
36 is that true? A. Radically superior.

Q. Now, have you ever tested the corresponding compound which has two carbon atoms in it? A. No.

Q. So, so far as you know that may even be worse than the cresyl, may it not? A. If I said I knew—so far as I knew, yes, but it would be contrary to all that I have learned.

Q. Well, would this be contrary to all you have learned; if you knew that cresyl was useful, wouldn't it be contrary to all you have learned to assume that the tri butyl would not be useful? A. It would be in effect, yes—I am sorry—

Q. Let's simplify it; wouldn't it be the fact that if you knew tricresyl was useful that it is probable at least that the other would also be useful? A. Yes.

Q. You would expect them to be useful, possibly more or less? A. Yes.

Q. But you would expect them to be useful? A. Yes, sir.

* * * * * * * * * *

37 Mr. Reynolds: I would like to offer for the defendant the folder which I handed to the Court, as defendant's exhibit number 1.

The Court: It may be admitted.

* * * * * * * * * *

III.

EXHIBITS.

Excerpts from Plaintiff's Exhibit No. 1.

93

Specification

To all whom it may concern:—

Be it known that I, Carl F. Prutton, a citizen of the United States and a resident of Cleveland Heights, County of Cuyahoga, and State of Ohio, have invented a new and useful Improvement in

"Lubricating Composition"

of which the following is a specification; the principle of the invention being herein explained, and the best mode in which I have contemplated applying that principle so as to distinguish it from other inventions.

94 This invention relates, as indicated, to lubricants and more particularly to a method of lubricating relatively moving metallic surfaces under conditions of extreme pressure as well as to methods of inhibiting corrosion of the surfaces and oxidation and sludging in the oil.

This application is a continuation in part of copending application Serial No. 737,070, filed July 26, 1934 and application Serial No. 760,038, filed Dec. 31, 1934.

By "extreme pressures" as herein used is meant pressures of or in excess of 10,000 pounds per square inch. Such pressures are encountered in hypoid gears, bearings, and contact surfaces of pistons and cylinders in internal combustion engines, as well, as in many other points of mechanical operation. It is a principal object of this invention to provide a method of lubricating and a method of improving the lubricating properties of lubricating oils and more particularly to provide a lubricating composition which will prevent seizure and scoring of relatively moving metallic surfaces where the pressure between such surfaces may be substantially in excess of 10,000 pounds per square inch, or where, for any reason, conditions of "thin-film" or "boundary" lubrication exist.

It is a well-known fault of mineral lubricating oils that they exhibit a tendency to form sludge upon standing or during use, this sludge lowering the fluidity of the oil and holding grit and other abrasive material very detrimental to the relatively moving parts being lubricated. Such oils become partially oxidized on contact with the atmosphere and sludging is also derived from such sources as the leakage of unconsumed fuel such as gasoline past the pistons and into the crank-case of an internal combustion engine.

A further object of this invention is, therefore, to provide a method of inhibiting the oxidation and sludging of mineral lubricating oils under conditions both of storage and use.

Another object of this invention is to provide a lubricating composition wherein, and a method of lubricating whereby, in addition to the above-named functions, corrosion of the metal parts is inhibited.

Other objects of this invention will appear as the description proceeds.

To the accomplishment of the foregoing and related ends, said invention, then, consists of the features hereinafter fully described and particularly pointed out in the claims, the following description setting forth in detail certain embodiments of the invention, these being illustrative, however, of but a few of the various ways in which the principle of the invention may be employed.

Broadly stated, this invention comprises a lubricating composition and method of lubricating under conditions of extreme pressure, wherein an addition agent comprised of an alkylated aromatic ester of an acid of phosphorus is employed in conjunction with a mineral oil base.

It has been found that addition agents of the type above defined are generally effective to inhibit oxidation and sludging in mineral oils and often markedly reduce corrosion of the bearing surfaces being lubricated. Because of the presence of the alkyl group or groups they are readily soluble in the mineral oil.

When very high pressures are to be encountered, it may be desirable to enhance the film strength-improving properties of the addition agent by halogenation, or more 96 specifically, chlorination. It is to be understood, of course, that the other halogens, namely fluorine, bromine, and iodine, are also effective in this respect. For maximum stability, the halogen is best attached to the benzenoid ring although, for various uses, it may satisfactorily and even preferably be attached to an alkyl group.

Since, in general, the loss of the addition agent by volatilization, will be less for compounds having a low volatility, the vapor pressure of the compounds should, for most purposes, be less than atmospheric pressure at temperatures up to 140° C. It is usually desirable, especially for use in internal combustion engines, that the vapor pressure be less than atmospheric at temperatures up to 170° C. In the case of compounds which are stable at temperatures up to their boiling points, this condition may be expressed by stating that the boiling point of the compound should, in general, be higher than 140° C., and for certain uses, such as internal combustion engines, higher than 170° C.

The following is a list of examples illustrating, but not intended to limit the type of addition agent contemplated by this invention:

I. The Alkylated Aryl Phosphates, e.g.
  tri-tertiary amyl phenyl phosphate
  tri-butyl phenyl phosphate
  tri iso-butyl phenyl phosphate
  tri-iso-propyl phenyl phosphite•

II. The alkylated Aryl Phosphites, e. g.
  tri-cresyl phosphite
  tri butyl phenyl phosphite
  tri iso-butyl phenyl phosphite
  tri iso-propyl phenyl phosphate

97 III. The alkylated Aryl Thiophosphates, e.g.,

tri butyl phenyl thiophosphate
tri iso-butyl phenyl thiophosphate
tri iso-propyl phenyl thiophosphate

One way of obtaining compounds of the type illustrated above is by condensing unsaturated chain compounds and more specifically, aliphatic hydrocarbons with aromatic esters of acids of phosphorus, such as tri-phenyl phosphate or tri-cresyl phosphate. A condensing agent of the type best illustrated by anhydrous aluminum chloride is employed.

The following are typical examples of suitable unsaturated compounds:

ethylene
propylene
isopropylene
n-butylene
isobutylene
polymerized butylenes
polymerized pentenes
polymerized hexenes
cracked gasoline
cracked kerosene
cracked waxes
cracked heavy oils
cracked petroleum

Any of the examples listed above may have its film strength-improving properties further enhanced by halogenation as noted supra. The halogen is believed to react chemically or physico-chemically with the metallic surfaces under conditions of extreme pressure to form an anti-fluxing and protective film thereon.

Lubricating compositions containing the addition agents of this invention, whether halogenated or unhalogenated, are particularly stable and resistant to high temperatures, properties which render them valuable for use as crankcase lubricants for internal combustion engines, including those of the aviation and Diesel types.

98 Alkylated aromatic esters of acids of phosphorus are preferred which contain at least one alkyl group having at least two, and still more desirably, at least four, carbon atoms in the chain, inasmuch as such compounds are more soluble in the mineral oil. This is important since it is often desirable to use relatively large proportions of the addition agent, up to about 10% by weight, based on the amount of mineral oil, when the advantage most desired is the increased film strength of the lubricant. In general, from about .5 to 5% is usually sufficient for this purpose and even less may occasionally be effective. The compounds with the longer alkyl groups also appear to exhibit better anti-oxidant properties and cause greater reduction in sludge-formation. When employed principally for the purpose of inhibiting oxidation of the lubricant or corrosion of the metallic surfaces lubricated, the proportion of addition agent employed will ordinarily be less than about 1% by weight, based on the amount of mineral oil, and from about .1% to about .5% will be the usual range although still smaller amounts may often be effective.

While mineral oil generally is the principal ingredient of the lubricant, it is not essential that it be the only ingredient other than the addition agent, provided that there should be no additional ingredient which is incompatible with such addition agent. It is within the contemplation of this invention to include, if necessary or desirable, such other addition agents as are commonly added to improve the viscosity index or cold test of the lubricant and a lubricating composition according to this invention which also has a separate oiliness-increasing agent has been found to be especially effective.

99 Other modes of applying the principle of my invention may be employed instead of the one explained, change being made as regards the materials employed, provided the ingredients stated by any of the following claims or the equivalent of such stated ingredients be employed.

I, therefore, particularly point out and distinctly claim as my invention:—

—1—

A lubricating composition comprising a mineral lubricating oil and a minor amount of an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

—2—

A lubricating composition comprising a mineral lubricating oil and a minor amount of an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms.

—3—

A lubricating composition comprising a mineral lubricating oil and a minor amount of an alkylated aromatic phosphite.

—4—

A lubricating composition comprising a mineral lubricating oil and a small percentage of an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

—5—

A lubricating composition comprising a mineral lubricating oil and a small percentage of an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms.

—6—

A lubricating composition comprising a mineral lubricating oil and a small percentage of an alkylated aromatic phosphite.

—7—

A lubricating composition comprising a mineral lubricating oil and from an effective amount to about 1% of an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

—8—

A lubricating composition comprising a mineral lubricating oil and an effective amount to about 1% of an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms.

—9—

A lubricating composition comprising a mineral lubricating oil and an effective amount to about 1% of an alkylated aromatic phosphite.

—10—

A lubricant for metallic bearing surfaces comprising a major proportion of a mineral lubricating oil, and a minor amount of an alkylated aryl ester of an acid of phosphorus, in which the alkyl radicle contains at least four carbon atoms.

101 Signed by me, at Cleveland, Ohio, this 22nd day of March, 1938.

Carl F. Prutton

OATH

State of Ohio
County Cuyahoga ss.

Carl F. Prutton, being duly sworn, deposes and says that he is the applicant herein, a citizen of the United States and a resident of Cleveland Heights, Cuyahoga County, Ohio; that he does verily believe himself to be the original, first and sole inventor of the Improvement in

"Lubricating Composition"

described and claimed in the foregoing specification, signed by him; that he does not know and does not believe that the same was ever known or used before his invention or discovery thereof, or patented or described in any printed publication in this or any foreign country before his invention or discovery thereof, or more than two years prior to this application, or in public use or on sale in this country for more than two years prior to this application; that his invention has not been patented to himself or others with his knowledge or consent in any foreign country on an application filed by him, or his legal representatives or assigns, more than twelve months prior to this application; and that no application for patent on same has been filed by him, or his legal representatives or assigns, in any foreign country.

CARL F. PRUTTON

Sworn to and subscribed before me, this 22nd day of March, 1938.

MABEL M. LYTTLE
(Seal) *Notary Public*

My Commission Expires Aug. 26, 1938

* * * * * * * * * *

150 U. S. Patent Office Board of Appeals Mailed Jun 30 1941

Paper No. 20

Appeal No. 36,558
RHJ

Hearing: June 11, 1941

In the United States Patent Office

Before the Board of Appeals

Ex parte Carl F. Prutton

Application for Patent filed March 23, 1938, Serial No. 197,732. Lubricating Composition.

Messrs. Oberlin, Limbach & Day for appellant.

This is an appeal from the action of the Primary Examiner finally rejecting claims 1 to 15.

Claim 1 is representative:

1. A lubricating composition comprising a mineral lubricating oil and a minor amount of an alkylated aromatic ester of an acid of phosphorus of the type obtained by con-

densing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

The references relied upon by the examiner are:

Graves,	2,005,619,	June 18, 1935,
Moran et al,	2,058,343,	Oct. 20, 1936,
Benning et al,	2,080,299,	May 11, 1937.

The examiner has held that appellant's application No. 737,070 cannot be relied upon to antedate the references because it:

"does not disclose and define the group of alkylated aromatic esters as compounds having a quality distinguishing them from the myriads of other classes enumerated therein."

151 The examiner does not hold that the earlier application does not contain a broad statement including the group of substances involved in this appeal; he does not state that sufficient examples of this group are not to be found in that application nor does he hold that the group is so broad as to cover substances which would be inoperative for the intended purpose.

We do not consider that an application which has a sufficiently broad disclosure to support claims to a group of substances cannot be relied upon for constructive reduction to practice of that group merely because that application does not point out that the matter of that group is patentably distinct from other matter disclosed in that application. It is believed, therefore, that the examiner's reason for holding that appellant's application 737,070 cannot be relied upon to antedate the reference, is not tenable.

The decision of the examiner is reversed.

BOARD OF APPEALS

W. L. THURBER
Examiner-in-Chief
F. P. EDINBURG
Examiner-in-Chief
F. J. PORTER
Examiner-in-Chief

Messrs. Oberlin, Limbach
& Day
909 Leader Bldg.
Cleveland, Ohio

June 30, 1941

* * * * * * * * * *

159 U. S. Patent Office Mailed Oct 12 1942

Div. 64 Room 267 JHM:NB Paper No. 27

Department of Commerce
United States Patent Office
Richmond, Va.

Applicant: Carl F. Prutton
Ser. No. 197,732
Filed Mar 23, 1938
For LUBRICATING COMPOSITION

Oberlin, Limbach & Day
909 Leader Bldg.
Cleveland, Ohio
Added art:

Malisoff	2,289,509	July 14, 1942 (filed 11/1/33)	252-54
Downing et al	2,285,853	June 9, 1942 (filed 2/23/34)	252-53

Claims 1 to 10 and 12 to 14 are rejected as being directly anticipated by the patents to Malisoff and Downing et al, each of which discloses a lubricating composition comprising a mineral lubricating oil and a minor amount of an aromatic or alkyl ester of phosphoric or phosphorous acid.

Claims 11 and 15 are rejected as being directly anticipated by the Malisoff patent which discloses halogenated aromatic esters of a phosphorus acid employed as a lubricating oil addition agent.

All the claims are rejected.

P. M. Nash
Examiner

Submitted Oct 8—1942 Supervisory Examiner

* * * * * * * * * *

166 U. S. Patent Office R Mailed Jan 15 1944

JHM/n Paper No. 31

Department of Commerce
United States Patent Office
Richmond, Virginia

On Appeal Before the Board of Appeals

In re application of
Carl F. Brutton
Serial No. 197,732
Filed Mar. 23, 1938
For: LUBRICATING COMPOSITION

Examiner's Statement

This is an appeal from the final rejection of claims 1, 2, 4, 5, 7, 8 and 10 to 15 (all the claims now in the case) as lacking invention over prior art.

The appealed claims are as follows:

1. A lubricating composition comprising a mineral lubricating oil and a minor amount of an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

2. A lubricating composition comprising a mineral lubricating oil and a minor amount of an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms.

4. A lubricating composition comprising a mineral lubricating oil and a small percentage of an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

5. A lubricating composition comprising a mineral lubricating oil and a small percentage of an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms.

167 7. A lubricating composition comprising a mineral lubricating oil and from an effective amount to about

1% of an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

8. A lubricating composition comprising a mineral lubricating oil and an effective amount to about 1% of an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms.

10. A lubricant for metallic bearing surfaces comprising a major proportion of a mineral lubricating oil, and a minor amount of an alkylated aryl ester of an acid of phosphorus, in which the alkyl radicle contains at least four carbon atoms.

11. A lubricating composition comprising a mineral lubricating oil and a minor amount of a halognated condensation product of an aromatic ester of a phosphorus acid with an unsaturated chain compound.

12. A lubricating composition comprising a mineral lubricating oil and a minor amount of tri-tertiary amyl phenyl phosphate.

13. A lubricant for metallic bearing surfaces comprising a major proportion of petroleum lubricating oil and a minor amount of an alkylated aryl phosphite having at least one alkyl group containing a plurality of carbon atoms.

14. A lubricant for metallic bearing surfaces comprising a major proportion of petroleum lubricating oil and a minor amount of an alkylated aryl phosphite having at least one alkyl group containing at least four carbon atoms.

15. A lubricant for metallic bearing surfaces comprising a major proportion of petroleum lubricating oil and a minor amount of a halogenated alkylated phosphite.

The following references are relied upon as anticipating the subject matter covered by the appealed claims.

Malisoff et al	2,289,509	July 14, 1942 (filed Nov. 1, 1933)
Downing et al	2,285,853	June 9, 1942 (filed Feb. 23, 1934)

168 SUBJECT MATTER INVOLVED

The present application relates, in general, to lubricating compositions and, more particularly, to lubricating compositions, the extreme pressure characteristics and stability of which have been improved by the addition of an alkylated aromatic ester of a phosphorus acid.

Ordinary lubricating oils are relatively ineffective when employed for the lubrication of moving surfaces under "extreme pressures" of the order of 10,000 pounds per square inch or more. Such oils also tend to oxidize in storage or under normal conditions of operation, resulting in the formation of sludge and acidic products of oxidation. These changes render the oils corrosive and reduce their efficiency as lubricants.

These defects have been previously recognized and it has now become conventional practice in the lubricating oil art, to add relatively small amounts of various types of addition agents to improve the extreme pressure characteristics of the oil and to stabilize the same against oxidative changes.

In the present instance applicant employs as an addition agent, an alkylated aromatic ester of an acid of phosphorus. It is stated that because of the presence of the alkyl group or groups, such compounds are readily soluble in mineral oil. Extreme pressure properties of these compounds may be improved by halogenation. For maximum stability it is indicated that the halogen is best attached to the benzenoid ring although for various uses, it may satisfactorily
169 and even preferably be attached to an alkyl group.

The phosphorus acid esters may be prepared by condensing unsaturated chain compounds, namely, aliphatic hydrocarbons, with aromatic esters of acids of phosphorus in the presence of a condensing agent, such as anhydrous aluminum chloride.

Esters containing at least one alkyl group having at least two or more carbon atoms in the chain are preferred due to the fact that such compounds are more soluble in mineral oil.

The proportion of addition agent ordinarily employed will be less than 1%, based on the amount of mineral oil, although from .5% or less up to 10.0% may be used. A list of esters said to be effective appears on pages 4 and 5 of the present specification.

Rejection of the Claims

The appealed claims may be conveniently divided into three groups, with respect to the addition agent, as follows:

Group I

Claims 2, 5, 8, 10, 12, 13 and 14.

Group II

Claims 1, 4 and 7.

Group III

Claims 11 and 15.

170

Group I

Group I, directed broadly to a lubricating oil containing a minor amount of an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms and specifically (claim 12) tritertiary amyl phenyl phosphate, is rejected as lacking invention over the patent to Downing et al. This patent discloses a lubricating composition comprising a lubricating oil having added thereto a minor amount of various esters of phosphorus acids, including esters of both phosphorus (phosphites) and phosphoric (phosphates) acids. Page 2, first column, lines 5 to 10 state:

"Also, when the term 'phosphorus acids' is employed hereinafter and in the claims, it will be understood that this term includes the various acids of phosphorus such as the various phosphoric, phosphorous and thio phosphorus acids".

Included in the specific compounds listed in the patent is tricresyl phosphate which is an alkylated aryl phosphate. The broader claims of Group I differ therefrom only in that the alkyl group is limited to one containing at least two carbon atoms as distinguished from the methyl derivatives listed in the patent. The primary reason for using alkyl groups of two or more carbon atoms is to insure adequate solubility of the compounds in the mineral oil, as pointed out at the top of page 6 of the present specification. Reference is made on the same page that compounds with longer alkyl groups also appear to exhibit better anti-oxidant properties and cause greater reduction in sludge formation but no data has been offered to support this general statement.

171 In regard to solubility, it is well known that the introduction of an alkyl group will promote solubility of a compound in mineral oil. Downing et al recognized this fact, pointing out at the bottom of page 1, second column that:

"Among the aliphatic esters, those containing a carbon chain of at least 8 carbon atoms have proved to be the most effective and of these, the mixed esters and mixtures of esters containing long chain aliphatic groups are generally preferred because of their greater solubility in lubricating oils and greases".

No invention is seen to reside in using the next higher member of a homologous series, particularly where the change is productive of no unusual or unexpected result. See Ex parte C. & L. (Case No. 114) 26 J. P. O. S. 68. Hence, to use the ethyl or even the butyl derivative as recited in claim 10, rather than the methyl derivative disclosed in the reference is not considered to represent a patentable contribution to the art.

Claim 12 is limited to a specific addition agent, namely, tri-tertiary amyl phenyl phosphate but there is no evidence of record to show that this compound produces a result different in kind from that obtained with the specific esters listed in the patent. Downing et al, moreover, refer to mixed tertiary amyl phenyl phosphates. It would appear to be immaterial with respect to the results secured whether the alkyl group is attached to the phenyl group or to the phosphorus through an oxygen atom.

GROUP II

The claims of Group II namely, 1, 4 and 7, are limited to esters prepared by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound. The particular manner in which the addition agents are 172 prepared is not regarded as material, in so far as the present composition claims are concerned. The addition agents prepared by condensing an unsaturated hydrocarbon with an aryl ester of a phosphorus acid are alkylated aryl esters of a phosphorus acid of the type recited in Group I. Moreover, the alkylation procedure employed by applicant is a conventional one in the art. If it were not conventional, the present disclosure would be insufficient since the conditions under which the condensation reaction is effected are not related.

GROUP III

Group III, embracing claims 11 and 15, are limited to compositions in which the alkylated aryl phosphorus esters are halogenated. These claims are rejected as lacking invention over the Malisoff patent which discloses lubricating oils containing minor amounts of halogenated alkylated esters of phosphorus acids, such as chlorinated tricresyl phosphate. The claims are regarded as unpatentable for the reasons fully set forth in the rejection of claims of Group I, supra.

CONCLUSION

The addition agents employed by applicant are regarded as an obvious variation of the agents disclosed in the references relied upon. Accordingly, it is submitted that patent protection on the appealed claims should be denied.

Respectfully submitted,

P. M. NASH
Examiner, Division 64

PMN.

* * * * * * * * * *

Affidavit of Albert K. Smith.

183 State of Ohio,
County of Cuyahoga, ss

Albert K. Smith, being first duly sworn, deposes and says that he is a resident of Shaker Heights, Ohio and is Vice President in Charge of Research and Development of The Lubri-Zol Corporation of Wickliffe, Ohio. Affiant has read and is thoroughly familiar with the specification in the application of Carl F. Prutton, Ser. No. 197,732, filed Mar. 23, 1938 wherein on page 3, the applicant after referring to the fact that his

> "invention comprises a lubricating composition and method of lubricating under conditions of extreme pressure, wherein an addition agent comprised of an alkylated aromatic ester of an acid of phosphorus is employed in conjunction with a mineral oil base"

continues with the statement that

> "It has been found that addition agents of the type above defined are generally effective to inhibit oxidation and sludging in mineral oils and often markedly reduce corrosion surfaces of the materials being inhibited."

Affiant has also noted the following further more specific reference by applicant to his preferred type of materials as given at the top of page 6 of the specification, viz:

> "Alkylated aromatic esters of acids of phosphorus are preferred which contain at least one alkyl group having at least 2 and still more desirably, at least four carbon atoms in the chain."

Applicant has been assigned the problem of determining the accuracy of the foregoing statements and to particularly make a comparison of the properties in mineral lubricating oils as concerns oxidation and corrosion of tri-ortho cresyl phosphate and tri-(t-butyl phenyl) phosphate.

184 In making these determinations, applicant, in the laboratory and with workers under his direction and supervision, selected the Continental oxidation test for the purpose of determining comparative data with respect to the two compounds.

The Continental oxidation test is identical with the standard and well known Indiana oxidation test except that a small piece of iron wire is included in the oil during the air blowing operation to serve as a catalyst so as to not only increase the rate of oxidation but to also more closely simulate actual operating conditions where the oil is generally at all times in contact with iron or contains finely divided iron in suspension.

The data thus determined is given on the attached sheet marked "Exhibit A" and in which the data in the several columns at the right-hand side of the sheet was that obtained when using the compositions named in the heading for each column. The comparative data included in the attached sheet shows that the two compounds tested are not at all alike with regard to the critical factors which were determined in this test. The comparative data with respect to oxidation and corrosion shows a marked superiority in effectiveness of tri-(tertiary butyl phenyl) phosphate as compared with tri cresyl phosphate.

Albert K. Smith

Sworn to and subscribed before me this 23rd day of January, 1945.

Lucille Jaeger,
Notary Public

Lucille Jaeger, Notary Public. My commission expires May 6, 1945

185 EXHIBIT "A".

COMPOSITION SAMPLE DESIGNATION	100% Oil W	99.75% Oil 25% Tri-O-Cresyl Phosphate X	99.67% of Oil 0.33% Tri-(t-butyl Phenyl) Phosphate Y
Continental Oxidation Test @ 50 hrs. 341° F., 10 Liters air per hr.			
Conradson Carbon			
On original sample	0.21	0.20	0.18
After 50 hrs.	0.74	0.64	0.36
Viscosity, SUS @ 210° F.			
On original sample	64.6	63.1	65.6
After 50 hrs.	66.0	69.5	69.5
% increase	2.78	10.1	5.94
Naphtha Insol.			
After 50 hrs.—%	0.081	0.132	0.090
Chloroform Soluble			
After 50 hrs—%	0.066	0.109	0.075
Appearance of Iron Springs			
After 50 hrs.	Tarnished	Tarnished	Unchange
Acid Number			
On original sample	0.05	0.05	0.05
After 50 hrs.	0.47	0.42	0.31

W is untreated oil
X is prior art additive
Y is Applicant's additive

* * * * * * * * * *

187 Paper No. 37

Department of Commerce
United States Patent Office
Richmond, Virginia

On Appeal Before the Board of Appeals

In Re Application of
Carl F. Prutton
Serial No. 197,732
Filed Mar. 23, 1938
For: Lubricating Composition

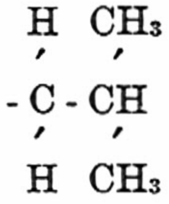
Report on Affidavit.

An affidavit by Albert K. Smith has been presented under the provisions of Rule 76 purporting to show that the addition agents covered by the appealed claims are superior anti-oxidants to those described in the prior art. Applicant, in general, employs alkylated aromatic esters of an acid of phosphorus, the alkyl group having at least two carbon atoms as distinguished from the corresponding compound disclosed in the prior art wherein the alkyl group has but a single carbon atom.

Data is presented comparing the results obtained by the Continental Oxidation Test with tri-o-cresyl phosphate (prior art) and tri-(t-butyl phenyl) phosphate. The latter compound is a tri alkyl phenyl phosphate wherein the alkyl groups contain four carbon atoms and hence the data is not applicable to the subject matter defined in claims 1, 2, 4, 5, 7, 8, 11, 13 and 15. These claims are sufficiently broad to cover compounds wherein the alkyl group contains two carbon atoms. To show that compounds of this type having an alkyl group of four carbon atoms exhibit
188 some advantage over the corresponding compound
having one carbon in the alkyl group does not mean that a compound having two carbon atoms in the alkyl group will show a similar advantage. The data presented is therefore regarded as inconclusive as to the subject matter covered by claims 1, 2, 4, 5, 7, 8, 11, 13 and 15.

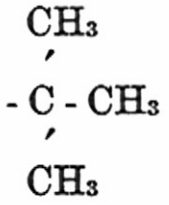
The data is also inconclusive as to the subject matter defined in claims 1, 2, 4, 5, 7, 8, 10 and 11 since these claims are sufficiently broad to cover both the phosphite and phosphate derivatives while the compound tested as set forth in the affidavit was a phosphate. Moreover, the additive recited in claims 13, 14 and 15 is a phosphite and the showing presented clearly is not applicable thereto.

The only claims wherein the additive is an alkylated aryl phosphate and wherein the alkyl group contains at least four carbon atoms is claim 12 but the compound recited therein, namely, tri-tertiary amyl phenyl phosphate, is not the compound selected as typical of those disclosed in the present case. In fact, it will be noted that the compound selected and tested for applicant as representative of those covered by the appealed claims is not specifically disclosed in the present specification. The compound tested was tri-(tertiary-butyl phenyl) phosphate, the location of the alkyl radical on the phenyl nucleus not being specified. The most closely related compound found in the present specification is tri iso-butyl phenyl phosphate. The structural formula of the alkyl substituent of this compound is



$$\begin{array}{ccc} & H & CH_3 \\ & | & | \\ - & C - & CH \\ & | & | \\ & H & CH_3 \end{array}$$

189 as compared with



$$\begin{array}{ccc} & CH_3 & \\ & | & \\ - & C - & CH_3 \\ & | & \\ & CH_3 & \end{array}$$

of the compound tested.

No explanation is offered as to why a compound not specifically disclosed in the specification was selected as a basis for comparison with the tri cresyl phosphate disclosed in the prior art.

In regard to the data submitted, tri-(t-butyl phenyl) phosphate does show some advantages over tri-o-cresyl phosphate but this must be largely discounted because 0.33% of the first mentioned compound was used as compared with 0.25% of tri-o-cresyl phosphate. No explanation is offered to show why 32% more of the compound selected as typical of those disclosed by applicant was used in carrying out the tests described in the affidavit.

CONCLUSION.

The affidavit is not considered to show that the compounds covered by the appealed claims are productive of a result patentably different from that obtained with the additives disclosed in the prior art.

P. M. Nash,
Examiner.

JHM/n

* * * * * * * * * *

192 Appeal No. 49,337 Paper #39
Hearing: January 24, 1945
In the United States Patent Office
Before the Board of Appeals
Ex parte Carl F. Prutton

Application for Patent filed March 23, 1938, Serial No. 197,732. Lubricating Composition
Oberlin, Limbach & Day for appellant.

This is an appeal from the final rejection of claims 1, 2, 4, 5, 7, 8 and 10 to 15.

Claim 1 is illustrative.

1. A lubricating composition comprising a mineral lubricating oil and a minor amount of an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.

The following references are relied upon:

Downing et al,	2,285,853,	June 9, 1942
Malisoff,	2,289,509,	July 14, 1942

The appealed claims relate to a lubricating composition comprising as an extreme pressure effective agent an alkylated aromatic ester of an acid of phosphorus. It is emphasized that the acid of phosphorus may be either of the phosphoric or phosphorous type. It is also a feature of each claim that the alkyl constituent of the compound must have at least two carbon atoms.

193 The examiner has broadly grouped the claims in three groups treating the claims 2, 5, 8, 10, 12, 13 and 14 of somewhat broader scope as lacking invention over the patent to Downing et al.

Downing et al disclose the same principle broadly of adding to lubricants an extreme pressure effective agent, among others—tricresyl phosphate. Downing et al emphasize that the ester may be formed of either phosphoric or phosphorous acid. The examiner concedes that tricresyl phosphate would not involve an alkyl group having at least two carbon atoms. It has only methyl groups attached to the aromatic radical. The examiner concludes that since one purpose of the alkyl radical is merely to make the extreme pressure agent more soluble in mineral oil lubricant that it is only a matter of choice of degree to select an alkyl group of two or more carbon atoms and further, that Downing et al recognized that fact in suggesting that they may use a compound having a carbon chain of at least eight carbon atoms. It appears, however, that it is uncertain whether this statement is intended necessarily to apply to the alkylated aromatic type but it is suggestive thereof. Downing et al disclose many examples, some of which are aliphatic only. However, we find among other examples in Downing et al mixed tertiaryamylphenyl phosphates and dilorol cresyl phosphate. In case of the former we would have five carbons in the alkyl chain and the term "lorol" is stated in the patent to mean in the range of coconut oils—page 2, lines 42-51. The patent to Downing et al therefore is regarded as actually satisfying the terms of the appealed claims in respect to the number of carbon atoms in
194 the combined alkylated aromatic radical. The rejection of the above group of claims upon Downing et al is affirmed.

Some showing has been attempted in the record as to minimizing sludge formation but the examiner has pointed out wherein the showing does not come within the original specific description of compounds. It is our opinion that so far as shown the same result would be accomplished by Downing et al.

The specific agent named in Claim 12 is not regarded as patentably different from the above example—tertiaryamylphenyl phosphate—page 3, line 69 of Downing et al.

Claims 1, 4 and 7 have been rejected as not developing any patentable matter in referring to the process of preparing the ester in these claims, apparently over the disclosure of Downing et al. The examiner holds that this states only an obvious mode of procedure in preparing the alkylated aromatics. We agree with this conclusion.

Claims 11 and 15 include limitation to a halogenated compound. The examiner rejects these claims upon the ground that halogenation of extreme pressure agents is known as in patent to Malisoff. That patent discloses chlorinated tricresyl phosphate. It is a matter of public knowledge in this relation that chlorinated compounds for this purpose have long been known. It appears merely an optional matter of choice in applicant's disclosure whether the present ester of phosphoric acid be chlorinated or not. In such situation and in view of Malisoff we agree with the examiner's conclusion that chlorination in claims 11 and 15 does not
serve to impart patentable matter to these claims. It
195 is observed that claim 15 does not require any aryl
constituent.

The discussion in the brief has been carefully considered without finding any reason for differing from examiner's conclusions.

The decision of the examiner is affirmed.

W. L. Redrow
Examiner-in-Chief
G. J. Porter
Examiner-in-Chief
E. F. Klinge
Examiner-in-Chief
Board of Appeals

March 2, 1945
Oberlin, Limbach & Day,
909 Leader Bldg.,
Cleveland, Ohio.

* * * * * * * * * *

205 Mailed Apr 4 1945 U. S. Patent Office Board of Appeals

Appeal No. 49,337 MWC Paper #41

In the United States Patent Office

Before the Board of Appeals

Ex parte Carl F. Prutton

Application for Patent filed March 23, 1938, Serial No. 197,732. Lubricating Composition.

Oberlin, Limbach & Day for appellant.

On Request for Reconsideration

The additional discussion has been carefully considered. It seems that the term "mixed tertiary amyl phenyl phosphates" should be taken as contended because of the term "mixed" and the plural form phosphates but the same is not true of the "dilorol cresyl phosphate". This should mean a compound where the two lorol radicals of fifteen carbon chains each are attached to the benzine ring of the cresyl radical rather than to the phosphoric acid radical. This is confirmed by the fact that it is of the same form of expression as found in appealed claim 12.

The showing of the affidavit in regard to tests is not persuasive of invention. There is only very general assertion (not stated how carefully measured or determined
206 as to be conclusive) that antioxidation was indicated. There is no showing of prevention of sludge formation or effectiveness of extreme pressure lubrication, prop-

erties, which seem to be the more important objects of the asserted invention. Further, there was no showing as to Downing's dilorol cresyl phosphate which is believed more relevant than his cresyl phosphate.

The petition has been considered but is denied with respect to making any change in our decision.

BOARD OF APPEALS

W. L. REDROW
Examiner-in-Chief

F. J. PORTER
Examiner-in-Chief

E. F. KLINGE
Examiner-in-Chief

April 4, 1945

OBERLIN, LIMBACH & DAY,
909 Leader Bldg.,
Cleveland, Ohio.

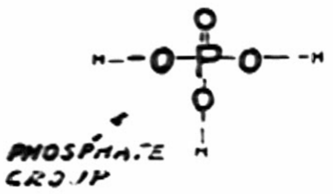
* * * * * * * * * *

The Lubri-Zol Corporation
vs.
Casper W. Ooms
C.A. 30,356
Plaintiff's Exhibit #2

Plf 2 A

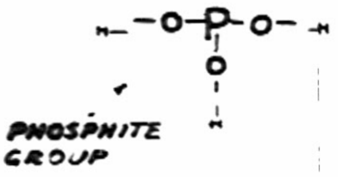
PHOSPHORIC ACID

Fig. 1



PHOSPHOROUS ACID

Fig. 2



METHANE

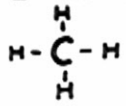


Fig. 5

FILED
JUN 5 1947
CHARLES E. STEWART, Clerk

METHYL GROUP

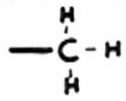


Fig. 6

BENZENE

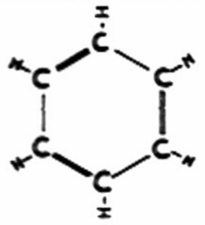


Fig. 3

BUTANE

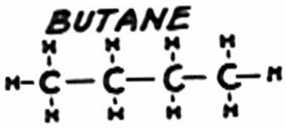


Fig. 7

BUTYLENE
AN UNSATURATED COMPOUND

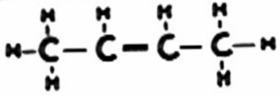


Fig. 8

PHENYL GROUP

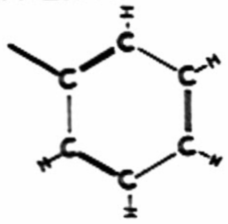


Fig. 4

BUTYL GROUP

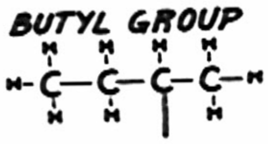


Fig. 9

THE LOROL* GROUP

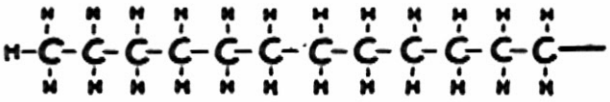


Fig. 10

* Lorol is defined in Downing et al patent No. 2,285,853 as a mixture of different chain length compounds. When there are 12 C's in the chain. . . as illustrated, the compound is a lauryl compound.

BEST COPY AVAILABLE
from the original bound volume

Plaintiff's Exhibit No. 3.

Extracts from the Flie of the Downing Patent No. 2,285,853.

215 Docket Division Mar 23 1938 U. S. Patent Office
Div. 64 Room 4708

Frederick Baxter Downing, Anthony Francis Benning and F. Willard Johnson

Lubrication

Filed February 23, 1934

Serial No. 712,610

Hon. Commissioner of Patents,
Washington, D. C.

Sir:

Please add the following new claim:

61. A lubricant for metallic bearing surfaces comprising a major proportion of a mineral lubricating oil and a minor amount of a mixed ester of an acid of phosphorus, which ester contains at least one radical of a phenol and at least one radical of an a-cyclic alcohol.

Remarks

Entrance of this amendment and allowance of new claim 61 is respectfully requested. This claim 61 corresponds to count (A) proposed in a motion to amend

216 80. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of a mixed ester of phosphoric acid, which ester contains at least one radical of a phenol containing only one benzene ring and at least one radical of a long-chain acyclic alcohol.

81. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of a mixed ester of phosphoric acid, which ester contains at least one radical of a phenol containing

only one benzene ring and at least one radical of a long-chain acyclic alcohol and each radical consists of the elements carbon and hydrogen.

82. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of dilorol cresyl phosphate.

REMARKS

Reconsideration and action on the merits of this application is respectfully requested.

All of the interferences, in which this application was involved, have now been terminated. Accordingly, it is in order to resume the Ex parte prosecution of this application.

By this amendment, all of the claims, except claims 46, 47, 60 and 61 have been cancelled. Thereby, all of the claims which have been lost in interferences, together with all claims which conflict with the claims being prosecuted in applicants' divisional applications have been cancelled. Thus, a clear line of division between the present application and applicants' divisional applications has been maintained.

217 parties attempted to make any claim corresponding to claims 73 to 76 or attempted to add any such claims to the interferences then pending, even though the party Downing et al. was claiming such subject matter. Under such circumstances, the party Downing et al. is presumed to be the first inventor of such subject matter under the provisions of Rule 116 and hence the disclosures appearing in the applications of Knutson et al., Caprio and Van Dijck are not available as references against the party Downing et al.

The acid esters, called for by applicants' claims, are more reactive than the neutral esters and tend to produce the lubricating film on the metallic surfaces more quickly. Accordingly, these acid esters are believed to be patentable over the neutral esters. Furthermore, claims 73 to 76 clearly come within allowed generic claims 71 and 72 and hence are clearly allowable therewith particularly in view of the decision in Ex parte Mitchell and Ex parte Dike et al. previously cited.

Applicants note that the Examiner has rejected claims 79 to 81 as being unpatentable over interference 77,319 and claims 79 to 82 as being unpatentable over interference 77,316. This rejection is also believed to be improper. The count of interference 77,319 is as follows:

An improved grease comprising a hydrocarbon lubricating oil and a thickening agent, having incorporated therein a small amount of tricresyl phosphate.

The tricresyl phosphate of this interference is not a mixed ester within any of these claims. Furthermore, the count required a thickening agent, whereas, applicants' claims do not. Claim 61 is generic to each of claims 79 to 82. This claim 61 constituted the count of interference 77,317 in which priority was awarded to Downing et al., Downing et al. prevailing over

* * * * * * * * * *

B

Pltf 4

FILED
JUN 5 1947
CHARLES E. STEWART, Clerk

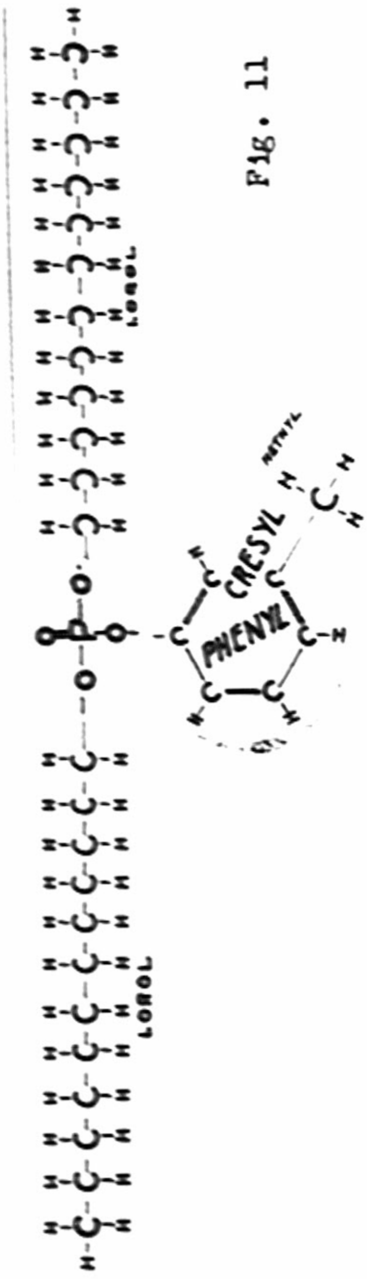


Fig. 11

DILOROL CRESYL PHOSPHATE

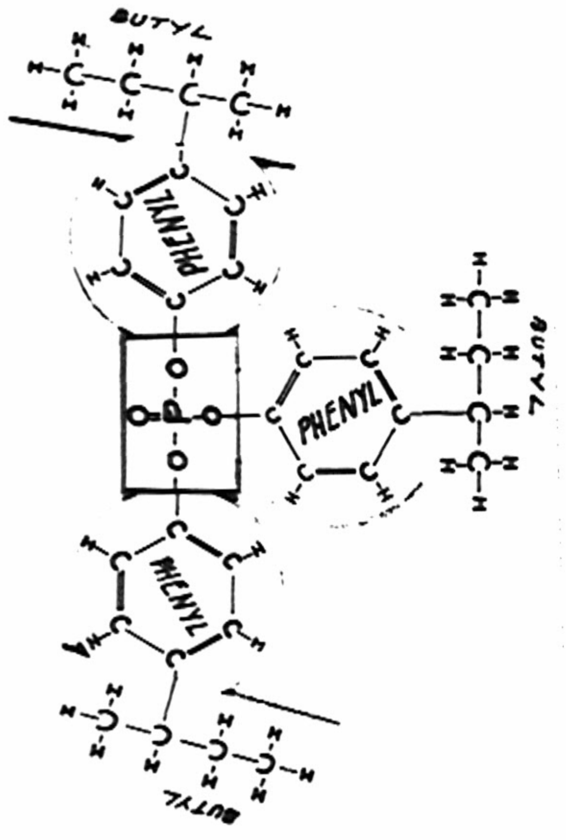


Fig. 12

TRI BUTYL PHENYL PHOSPHATE

The Lubri-Zol Corporation
vs.
Casper W. Ooms
C.A. No. 30,356
Plaintiff's Exhibit #4

219

BEST COPY AVAILABLE
from the original bound volume

DEFENDANT'S EXHIBIT NO. 1.

Patented June 9, 1942 **2,285,853**

UNITED STATES PATENT OFFICE

2,285,853

LUBRICATION

Frederick Baxter Downing, Carneys Point, Anthony F. Benning, Penns Grove, and Frank Willard Johnson, Pennsville, N. J., assignors to E. I. du Pont de Nemours & Company, Wilmington, Del., a corporation of Delaware

No Drawing. Application February 23, 1934, Serial No. 712,610

15 Claims. (Cl. 252—53)

FILED
JUN 5 1947
CHARLES E. STEWART, Clerk

This invention relates to lubrication and particularly to the improvement of extreme pressure lubricating characteristics by the addition of assisting agents.

The advantages of a high pressure lubricant become apparent when the present trend in design of automotive and other machine parts, and the increased strength of metal parts, due to the use of alloy steels, is considered. The pressures ordinarily found in well lubricated journal bearings do not exceed 2,000 lbs./sq. in. and for conditions such as this a film of heavy oil can be expected to remain between the rubbing surfaces. When gears are considered, where the contact between surfaces is of very small width, the bearing pressures often reach values as high as 25,000 lbs./sq. in. Under such extreme pressure it is unlikely that any oil or grease can be obtained which will be viscous enough to prevent metallic contact. The result of the lack of a lubricating film between the rubbing surfaces results in scoring and scuffing of the gears.

Failure to maintain lubricant films on idle bearing surfaces is now well recognized as the cause of 70 to 80% of the wear occurring on cylinder walls of automotive and other internal combustion engines. When an engine is stopped, the oil film on the vertical surfaces soon drains off and, when the machine is again started up, an appreciable time elapses before the circulation provides oil for the formation of new lubricant films. An assistant which, when added to the motor oil, will prevent the separation of the oil film from the metal should prevent a large part of the wear now ocurring.

Prior to this invention various assistants have been added to oils to improve their lubricating properties. Among such assisting materials which have been used are sulfur, sulfur chloride, sulfurized oil or oil containing naturally occurring sulfur, various heavy metal soaps and aluminum soaps, castor oil and, recently, aliphatic and aromatic halides. While some of these assistants, such as the sulfur and the organic halides improve the lubricating properties of the oil to a great extent, they have not proved to be satisfactory for use for the reason that they are corrosive or give rise to corrosive agents by hydrolysis or oxidation. Also, this corrosive action is greatly intensified by the presence of small amounts of water. Some of the alkyl halides are volatile and, hence, are soon lost. Other assistants such as the alkali and heavy metal soaps have proved to be of little value in improving the lubricating properties of the oils.

An object of this invention is to provide materials, which, when brought into contact with metal bearing surfaces, will form a lubricating film between such bearing surfaces, which lubricating film will adhere to the metal surfaces and resist high pressures. A further object of this invention is to provide materials which, when added to lubricating media improve their properties, particularly their adhesiveness and resistance to high pressure. A still further object is to provide a method for retarding the wearing, scuffing and seizing of bearing surfaces, adapted to operate at high bearing pressures, by contacting such surfaces with materials which will form adherent films on such bearing surfaces, which films will not break down under high bearing pressures. Other objects are to provide new compositions of matter and to advance the art. Still other objects will appear hereinafter.

These objects may be accomplished in accordance with our invention which comprises contacting metal bearing surfaces with an organic ester of a phosphorus acid. When such esters are brought into contact with a metal bearing surface they adhere to such surface forming a noncorrosive lubricating film which will retard the wear of the bearing surfaces, will withstand high bearing pressures and will prevent metallic contact of the bearing surfaces under high bearing pressures. The films have exceptional lubricating properties at all pressures but are particularly valuable at high bearing pressures and at high rubbing speeds. Such films will not be washed from the bearing surfaces by water, oil or organic solvents such as gasoline, naphtha and the like.

The organic esters of our invention may be employed by themselves as lubricants. However, it will generally be found to be desirable, for economical reasons, to employ them in a liquid carrying medium or a grease. The liquid carrying medium may be any liquid in which the organic esters may be dissolved, dispersed or emulsified. It will be preferred to employ a liquid which is noncorrosive to metal in the presence of the organic ester. Among the liquids which have been successfully employed are vegetable oils, kerosene, naphtha, alcohols, water, and lubricating oils and greases. For lubricating gears and similar bearing surfaces, it will generally be desirable to employ lubricating oils and greases as the carrying media.

Among the compounds which we have found to be particularly effective for our purpose are the aryl and aliphatic esters of phosphoric and phosphorous acids. Among the aliphatic esters, those containing a carbon chain of at least 8 carbon atoms have proved to be the most effective and of these, the mixed esters and mixtures of esters containing long chain aliphatic groups are generally preferred because of their greater solubility in lubricating oils and greases.

BEST COPY AVAILABLE
from the original bound volume

When the expression "long chain aliphatic (or alkyl)" is employed hereinafter and in the claims, it will be understood that such expression means aliphatic or alkyl groups containing 8 or more carbon atoms. Also, when the term "phosphorus acids" is employed hereinafter and in the claims, it will be understood that this term includes the various acids of phosphorus such as the various phosphoric, phosporous and thio phosphorus acids. The term "bearing surfaces" as employed herein and in the claims will be understood to mean surfaces which mutually carry a load and move relatively to each other.

The organic esters of the phosphorus acids of our invention may be employed as the free esters or as their alkali salts such as the sodium, potassium, ammonium and organic amine salts. The addition of as small amount as 0.05% to an oil will have a noticeable effect. However, for practical use, it will generally be found desirable to use at least 0.2%. Further, the amount added to the oil or other carrier will be largely dependent upon the machinery in which it is applied and the area of the metal surfaces with which it will be brought into contact.

The method of testing our compounds was that devised by J. O. Allmen (Oil and Gas Journal, 30, 109, 1931). This method consists of running a ¾" diameter drill rod between two halves of a split bushing which is maintained stationary. The load on the bushing is controllable and provision is made for measuring the torque developed by the friction of the lubricant film. A hydraulic system for increasing the loading on the bushing until the oil film breaks and the metal seizes is provided. The rubbing speed is about 50 feet per minute and the method of loading is gradual, one weight being added to the loading lever each ten seconds. Each weight added to the loading lever increases the pressure on the bushing by about 125 lbs. The machine provides for beam loadings up to 20 weights which corresponds to a pressure of 20,000 lbs./sq. in. on the full projected area of the drill rod. The bearing surface of the bushing is cut to a diameter 0.007 inch larger than the drill rod so that, before any wear occurs, the actual bearing surface is a line. As wear occurs, the bearing surface widens but seldom covers the bushing. After a test, the width of the bearing scar can be measured and an approximate value for the actual bearing pressure obtained. The values given in the following examples represent the calculated actual bearing pressures which were reached in the tests without failure of the film. These values represent film strength or film resistance.

When subjected to the above test, a good grade of paraffin oil will withstand a pressure of only 3 to 5,000 lbs./sq. in. When an oil containing sulfur is tested by the same method, such oil will show a film strength of about 20,000 lbs./sq. in. and will give a torque reading of over 4.0 lbs. ft. at this load.

In order to more clearly illustrate our invention and the advantageous results to be obtained thereby the following examples are given:

Example 1

Five-tenths percent of dilauryl phosphate $(C_{12}H_{25})_2HPO_4$ is added to a medium viscosity (S. A. E. 30) mineral oil and the whole warmed slightly until the ester has dissolved. When tested by the method hereinbefore described, this oil shows a film resistance of more than 55,000 lbs. per sq. in. and the friction developed is 2.0 to 2.2 lb. ft. When larger amounts of the phosphate up to 4% were added to the same oil the same results are obtained.

Example 2

An oil mixture was made up as in Example 1, except that 1.0% of dicetyl phosphate was employed in place of the dilauryl phosphate. The load carried on test is more than 55,000 lbs. per sq. in. and the torque is 2.0 lb. ft.

Example 3

Five percent of tricresyl phosphate was added to a medium viscosity (S. A. E. 30) mineral oil as in Example 1. The load carried on test was more than 25,000 lbs. per sq. in. and the torque developed was 3.5 lb. ft.

Example 4

1.0% of dicyclohexyl phosphate was added to a medium viscosity mineral oil (S. A. E. 30). The load carried was 20,000 lbs. per sq. in. and the torque developed was 1.4 lb. ft.

Example 5

1% of dioleyl phosphate was added to a mineral oil (S. A. E. 30). The load carried was 45,000 lbs. per sq. in. and the torque was 2.0 lb. ft.

Example 6

A lubricant made by dissolving 1% of dilauryl phosphate in a heavy oil (600 W) withstood a pressure of more than 52,000 lbs. per sq. in. and developed a torque at this pressure of 2.2 lb. ft.

Example 7

A lubricant was prepared as described in Example 1, except that 1.0% of a mixture of di- and tri-lorol phosphites was employed in place of the dilauryl phosphate. The load carried on test was 51,000 lbs. per sq. in. and the torque at this load was 2.3 lb. ft.

The term lorol as employed in this and other examples and throughout the specification and claims indicates a mixture of primary normal aliphatic alcohols of 8 and 10 carbon atoms which are obtained by fractionation of the alcohols resulting from the reduction of cocoanut and/or palm kernel oils. These alcohols had a boiling range of 140 to 195° C. at 50 mm. Accordingly, the term "lorol phosphites" means the phosphorous ester of such alcohols.

Example 8

One part of triphenyl phosphite $((C_6H_5)_3PO_3)$ mixed with 100 parts of a lubricating oil withstood a bearing pressure of 48,000 lbs./sq. in. The friction torque was 3.2 lb. ft.

Example 9

A medium viscosity mineral oil (S. A. E. 30) containing 1% of trinaphthyl phosphate carried a load of 21,000 lbs. per sq. in., the torque was 2.2 lb. ft.

Example 10

A mixture was made up of one part dilorol phosphate and 99 parts of solvent naphtha. Under test, this lubricant withstood a pressure of more than 45,000 lbs. per sq. in. with a friction torque of 2.2 lb. ft.

Example 11

One part of dilorol phosphate was mixed with 99 parts of lorol alcohol, comprising a mixture of primary aliphatic alcohols containing 8 to 10 carbon atoms. This mixture withstood a load of

BEST COPY AVAILABLE
from the original bound volume

32,000 lbs. per sq. in. developing a torque of 2.1 lb. ft. Under the same conditions of test, a lorol alcohol film, in the absence of the phosphate, will break with less than 4,000 lbs. pressure per sq. in.

Example 12

A mixed phosphate ester of lorol and ocenol tested alone withstood a bearing load of 45,000 lb. per sq. in. with a torque of 2.1 lb. ft.

Ocenol comprises a mixture of primary aliphatic alcohols containing 12 to 18 carbon atoms, having an iodine number of about 50 and is mostly oleyl alcohol.

Example 13

5.0% of a naturally occurring phosphate ester, lecithin, was added to a lubricating oil as in Example 1. On test, this mixture withstood a pressure of 52,000 lbs. per sq. in. and developed a torque of 3.0 lb ft.

Example 14

One part of dilorol phosphate was mixed with 200 parts castor oil. This mixture withstood a pressure of 60,000 lbs. per sq. in. The friction torque was 2.4 lb. ft.

Example 15

One part of dilorol phosphate was emulsified in 100 parts of water containing a small amount of caustic soda. This emulsion withstood a pressure of 32,00 lbs. per sq. in. with a friction torque of 1.9 lb. ft.

Example 16

One part of the a-naphthylamine salt of dilorol phosphate was mixed with 100 parts of lubricating oil. On test, this lubricant withstood a pressure of over 55,000 lbs. per sq. in., at which load the friction torque was 2.4 lb. ft.

Example 17

One part of dilauryl dithio phosphate was compounded with 100 parts of lubricating oil. The presure carried was 34,000 lbs. per sq. in. The torque was 2.1 lb. ft.

The above examples merely illustrate the results obtained with a few of the compounds of our invention. Other compounds coming within our invention which we have found to be effective for our purpose are:

Di-ethyl phosphate
Tri-ethyl phosphate
Mixed cyclohexyl-cylohexyl phosphates
Tri-ocenol phosphate
Di-octyl phosphate
Mixed mono- and di-lorol phosphates
Mixed octyl lauryl phosphate (di-ester)
Tri-methylricinoleyl phosphate
Lauroxy-ethanol phosphate
The monophosphate of the diglyceride obtained by partial hydrolysis of linseed oil
Mixed phosphate esters of hydroxy stearic acid
Cetyl-pyridinium phosphate
Di-phenyl phosphate
Di-cresyl phosphate
Tri-nitrophenyl phosphate
Tri-hexoxyphenyl phosphate
Mixed tertiaryamylphenyl phosphates
Mixed dodecahydro-diphenylol-propane phosphates
Mixed mono- and di-decahydro-b-naphthyl phosphates
Mixed tetrahydro-b-naphthyl phosphates
✓ Dilorol cresyl phosphate
Dibutylamine salt of mixed phosphates of lorol
Benzylamine salt of mixed phosphates of lorol
Amylamine salt of mixed phosphates of lorol
Mixed mono- and di-butyl phosphates

The mono- di- and tri-esters corresponding with those heretofore mentioned may also be mentioned. Esters of phosphorus acids may be obtained by the treatment of a mineral or natural oil with phosphorus halides and a catalyst such as aluminum chloride. Phosphate and phosphite esters can also be obtained by the treatment of oils containing ethylene linkages or hydroxyl groups with phosphorus halides, oxy halides, oxides, sulfides, etc. The resultant esters will be found to be effective for our purpose and come within the scope of our invention.

From the preceding description it will be apparent that the organic esters generally of the phosphorus acids are lubricants for metallic surfaces and suitable assistants to be added to lubricating oils, greases and other liquids to form lubricating compositions for metallic surfaces and which will be effective at extreme pressures.

The compounds of our invention, when brought into contact with metal bearing surfaces, apparently form an adherent film thereon which film will withstand high bearing pressures without breaking down and thus will prevent metallic contact of the bearing surfaces. When incorporated in an oil or grease, our compounds cause the oil and grease to adhere more firmly to the bearing surfaces. This is particularly desirable and advantageous characteristic of our compounds, particularly adapting them for use where the bearing surfaces are vertical and an ordinary oil tends to drain therefrom, as in the cylinders of internal combustion engines. Furthermore, the film produced by our compounds remains on the bearing surfaces even after the oil or other carrying media has been drained off and continues to exert its effect until worn off. Such film will not be removed by washing with water, gasoline, solvent naphtha and the like.

Our compounds are non-corrosive and, in fact, some of them will inhibit corrosion of metal by water. Our compounds are effective where the rubbing speeds are high as well as where they are low as in gears.

The fact that our compounds retain their extreme pressure lubricating characteristics in liquids other than oils and greases renders them particularly adapted for use as cutting oils, particularly in water emulsion. Our compounds may be employed in other liquids than those mentioned in the examples. For instance, they may be employed in benzene, gasoline, diphenyl, diphenyl oxide and any other liquid which is noncorrosive to metal and in which our compounds may be dissolved, dispersed or emulsified.

The use of cutting oils in machining operations is well known. The cutting fluid has two functions; the first to provide a lubricant to reduce the friction between the cutting tool and the work, and the second to provide a means for cooling the work.

The general practice is to use either an oil or a dispersion of oil in water. The oils used vary considerably depending on the particular operation and may vary from a light mineral oil to a heavy mixture of mineral and animal oils. The water suspensions are usually made up from oils by the addition of dispersing agents of the Turkey red oil type. Recently the practice of adding sulfur or sulfur compounds has become common.

BEST COPY AVAILABLE

Die shaping of sheet metals and drawing of sheets and wires and tubes has also recently been improved by the use of a lubricant between the contacting surfaces.

Cutting oils are now generally made up by compounding about 30 parts of lard oil, one to five parts of sulfur, two to ten parts of a petroleum sulfonate or Turkey red oil and about 60 parts of mineral oil. This mixture is then used undiluted or dispersed in water which improves its cooling properties.

The lard oil, of course, breaks down under the high temperature conditions of use resulting in the formation of disagreeably odorous materials. The presence of sulfur, as in motor and gear lubricants, is a possible source of corrosive material necessitating the careful washing of machined parts before use. These oils have also been given considerable trouble from infection of the workmen which is only partially eliminated by the incorporation of a bactericide in the oil.

Wire drawing also requires an extreme pressure lubricant. Several types are in use but essentially they are the same as the ordinary cutting oils.

The pressures between the rubbing surfaces during all machining operations are obviously very high as they result in deformation of the metal. This is, therefore, another problem of extreme pressure lubrication.

Our compounds may be used in water, oil or other suitable media as cutting oil compositions with the attending elimination of the odors and corrosiveness which are objectionable properties of most prior cutting oils. They will be effective at very low concentrations in oil and can be used in water directly without the use of an oil medium.

Other improvements similar to those observed on bearing lubrication should also result from such use. Decreased friction, safety from corrosion, and improved quality of the work are important results of their use.

The compounds of our invention are in general well known and the methods for making them are also well known and described in the literature. These methods generally comprise the treatment of alcohols, unsaturated aliphatic compounds or phenols with phosphorus chlorides or with phosphorus oxy chlorides or oxides. Accordingly, a detailed description of the methods to be employed for making the compounds referred to in this application is believed to be unnecessary.

While we have disclosed specific embodiments of our invention, it will be readily apparent to those skilled in the art that many modifications and variations may be made therein without departing from the spirit of our invention. Accordingly, the scope of our invention is to be limited solely by the appended claims construed as broadly as is permissible in view of the prior art.

We claim:

1. A lubricant for metallic bearing surfaces consisting of a major proportion of a petroleum lubricating oil and a minor amount of an acid ester of a sulfur-free acid of phosphorus.

2. A lubricant for metallic bearing surfaces comprising a major proportion of a mineral lubricating oil and a minor amount of a mixed ester of an acid of phosphorus, which ester contains at least one radical of a phenol and at least one radical of an a-cyclic alcohol.

3. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of an aromatic acid ester of a phosphorus acid.

4. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of an aromatic acid ester of a phosphorus acid in which each aromatic group contains a single benzene ring.

5. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of a diaromatic acid ester of phosphoric acid.

6. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of a diaryl acid ester of phosphoric acid in which each aryl group contains a single benzene ring.

7. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of acid dicresyl phosphate.

8. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of acid diphenyl phosphate.

9. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of a mixed ester of an acid of phosphorus, which ester contains at least one radical of a phenol containing only one benzene ring and at least one radical of a long-chain acyclic alcohol.

10. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of a mixed ester of an acid of phosphorus, which ester contains at least one radical of a phenol containing only one benzene ring and at least one radical of a long-chain acyclic alcohol and each radical consists of the elements carbon and hydrogen.

11. A lubricant for metallic bearing surfaces comprising a major proportion of a mineral lubricating oil and a minor amount of a mixed ester of phosphoric acid, which ester contains at least one radical of a phenol and at least one radical of an acyclic alcohol.

12. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of a mixed ester of phosphoric acid, which ester contains at least one radical of a phenol containing only one benzene ring and at least one radical of a long-chain acyclic alcohol.

13. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of a mixed ester of phosphoric acid, which ester contains at least one radical of a phenol containing only one benzene ring and at least one radical of a long-chain acyclic alcohol and each radical consists of the elements carbon and hydrogen.

14. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of dilorol cresyl phosphate.

15. A lubricant for metallic bearing surfaces comprising a major proportion of a hydrocarbon lubricating oil and a minor proportion of an organic acid-ester of a phosphorus acid.

FREDERICK BAXTER DOWNING.
ANTHONY F. BENNING.
F. WILLARD JOHNSON.

BEST COPY AVAILABLE

from the original bound volume

CERTIFICATE OF CORRECTION.

Patent No. 2,285,853. June 9, 1942.

FREDERICK BAXTER DOWNING, ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 4, first column, line 19, strike out the word "been"; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 18th day of August, A. D. 1942.

Henry Van Arsdale,
(Seal) Acting Commissioner of Patents.

Patented July 14, 1942 **2,289,509**

UNITED STATES PATENT OFFICE

2,289,509

LUBRICANT

William M. Malisoff, Philadelphia, Pa., assignor
to The Atlantic Refining Company, Philadelphia, Pa., a corporation of Pennsylvania

No Drawing. Application November 1, 1933,
Serial No. 696,150

11 Claims. (Cl. 252—54)

The present invention relates to the art of lubrication, and more particularly to the lubrication of surfaces engaging under extreme pressure, as for example, the rubbing surfaces of hypoid gears, free wheeling transmissions and speed reducers.

The general tendency in the design of modern machinery has been toward a higher ratio between power and "dead weight." This is especially true in the automotive industry, and in recent years certain types of gears and other mechanisms have been developed with the operating pressures on the working surfaces so high that ordinary mineral oil lubricants will not provide sufficient lubrication for satisfactory operation.

Heretofore it has been though that lubrication consists in maintaining a film of oil between the rubbing surfaces, thereby preventing them from coming into contact with one another and thus preventing wear. That this condition exists in well lubricated bearings is well known, but this conception of lubrication does not apply to highly loaded gears. In well lubricated bearings the loads rarely exceed 2000 lbs. per sq. in. projected area and the rubbing speeds are generally high enough to maintain a film of oil which separates the rubbing surfaces. In automobile gears, the pressures between gear teeth reach very high values and even the most viscous oils or greases cannot be retained between the surfaces of the teeth in a sufficiently thick film to prevent metal to metal contact, particularly when operating temperatures of 210° F. or higher are commonly encountered.

It has been known that compounded lubricants such as, for example, mineral oils containing fatty oils, fatty acids, metallic soaps or sulfur, possess lubricating qualities which render them suitable for use under high operating pressures. These compounded oils function satisfactorily under conditions which would cause failure of an unblended mineral oil, if used alone. It is believed that the successful use of such compounded oils depends upon the adsorption and reaction or union of certain components of these oils with the metal surfaces whereby a film of metallic compound, such as for example, iron sulfide is formed. It appears that such a film or plating has a low coefficient of friction and that satisfactory operation of heavily loaded bearings or gears depends upon the formation and maintenance of such a film, and not upon the retaining of a film of oil between the bearing surfaces. The hydrocarbon oil constituent of an extreme pressure lubricant serves primarily to remove frictional heat, to wash away any solid particles which may result from wear, and to prevent oxidation of the engaging surfaces.

I have discovered that the esters of the phosphorus acids and more particularly the halogenated esters of said acids, when admixed with hydrocarbon oils, are of special utility in the field of extreme pressure lubrication. The esters contemplated may contain alkyl radicals or aryl radicals, or a combination of alkyl and aryl radicals. The alkyl radicals may be represented, for example, by the radicals methyl, methylene and methine and their homologues, while the aryl radicals may be phenyl, naphthyl and anthracyl and their homologues. The akyl esters may be represented, for example, by tri-butyl phosphate, the aryl esters by tri-phenyl phosphate and the mixed esters by mono-butyl di-phenyl phosphate. I have found that halogenation of the esters may be effected under various conditions of reaction time, temperature and pressure, and in the presence or absence of a catalyst. The halogenated products may contain one or more halogen atoms to the molecule, depending upon the operating conditions and the quantity of halogen desired in the final product.

In accordance with my invention, I produce an extreme pressure lubricant which is of uniform composition, stable, non-corrosive and which will lubricate gears, bearings and similar rubbing surfaces under heavy loads in a satisfactory manner. In preparing my lubricant, I add to a suitable mineral oil an ester of a phosphorus acid in quantity sufficient to improve the lubricating value of the oil to any desired extent, depending upon the operating conditions under which the lubricant shall be used. I have found that the quantity of ester required, in general, does not exceed substantially 10% by weight of my composition. Quantities of ester as small as 3%, or even 1% or less, in certain instances, have been found to improve lubricating oils to a satisfactory extent. In preparing my lubricant, I may obtain a homogenous solution of ester in mineral oil by agitating the mixture at normal or elevated temperatures, or I may dissolve the ester in a suitable solvent and add the resulting solution to the oil, thereafter removing the solvent by vaporization.

Typical examples of my improved extreme pressure lubricant are as follows:

1. 97 parts of a Mid-Continent lubricating oil having a Saybolt universal viscosity at 210° F. of 158 seconds and an A. P. I. gravity of 22.9° was

BEST COPY AVAILABLE
from the original bound volume

admixed with 3 parts of tri-cresyl phosphate and a homogeneous solution was obtained. Upon testing this lubricant in a Timken lubricant tester, a pressure of 33,000 lbs./sq. in. was required before scoring of the test bearing was obtained, whereas the unblended lubricating oil scored at 10,000 lbs./sq. in. pressure.

2. 90 parts of a Pennsylvania cylinder oil having a Saybolt universal viscosity at 210° F. of 203 seconds and an A. P. I. gravity of 25.4° was admixed with 10 parts of tri-butyl phosphate and a homogeneous solution was obtained. Upon testing this lubricant in a Timken lubricant tester, a pressure of 26,000 lbs./sq. in. was required before scoring of the test bearing was obtained, whereas the unblended cylinder oil scored at a pressure of 10,000 lbs./sq. in.

3. Tri-cresyl phosphate was chlorinated at a temperature of from 75° F. to 122° F. in the absence of a catalyst; the resulting product was then air blown to remove hydrochloric acid and thereafter heated to 575° F. for approximately ½ hour and then cooled. The resulting chlorinated tri-cresyl phosphate contained 3.1% of chlorine. One part of this chlorinated ester was admixed with 99 parts of a Pennsylvania lubricating oil having a Saybolt universal viscosity at 210° F. of 158 seconds and an A. P. I. gravity of 22.9°, and a homogeneous solution was obtained. Upon testing this lubricant in a Timken lubricant tester, a pressure of 30,000 lbs./sq. in. was required before scoring of the test bearing was obtained, whereas the unblended lubricating oil scored at a pressure of 10,000 lbs./sq. in.

4. Tri-cresyl phosphate was chlorinated at a temperature of from 75° F. to 140° F. in the presence of a small quantity of iodine as catalyst; the resulting product was then washed with a dilute caustic soda solution and water, and was thereafter dried by air blowing at 285° F. The resulting chlorinated ester contained 9.7% of chlorine. Ten parts of this chlorinated ester was admixed with 90 parts of a Pennsylvania cylinder oil having a Saybolt universal viscosity at 210° F. of 144 seconds and an A. P. I. gravity of 26.6° and a homogeneous solution was obtained. Upon testing this lubricant in a Timken lubricant tester, a pressure of 24,000 lbs./sq. in. was required before scoring of the test bearing was obtained, whereas the unblended lubricating oil scored at a pressure of 10,000 lbs./sq. in.

5. Tri-cresyl phosphate was chlorinated at a temperature of from 75° F. to 140° F. in the presence of a small quantity of iodine as catalyst; the resulting product was then washed with a dilute caustic soda solution and water, and was thereafter dried by air blowing. The resulting chlorinated ester contained 20.1% of chlorine. Three parts of the chlorinated ester was admixed with 97 parts of a Mid-Continent lubricating oil having a Saybolt universal viscosity at 210° F. of 158 seconds and an A. P. I. gravity of 22.9° and a homogeneous solution was obtained. Upon testing this lubricant in a Timken lubricant tester, a pressure of 23,000 lbs./sq. in. was required before scoring of the test bearing was obtained, whereas the unblended lubricating oil scored at a pressure of 10,000 lbs./sq. in.

6. Tri-butyl phosphate was chlorinated at a temperature of from 75° F. to 120° F., no catalyst being present, and the resulting product was then neutralized by filtration through brucite ($Mg(OH)_2$). The chlorinated ester was found to contain 5.9% chlorine. Three parts of the chlorinated ester was admixed with 97 parts of a Mid-Continent lubricating oil having a Saybolt universal viscosity at 210° F. of 158 seconds and an A. P. I. gravity of 22.9° and a homogeneous solution was obtained. Upon testing this lubricant in a Timken lubricant tester, a pressure of 20,000 lbs./sq. in. was required before scoring of the test bearing was obtained, whereas the unblended lubricating oil scored at a pressure of 10,000 lbs./sq. in.

7. 97 parts of a Mid-Continent lubricating oil having a Saybolt universal viscosity at 210° F. of 158 seconds and an A. P. I. gravity of 22.9° was admixed with 3 parts of tri-phenyl phosphate and a homogeneous solution was obtained. Upon testing this lubricant in a Timken lubricant tester, a pressure of 18,000 lbs./sq. in. was required before scoring of the test bearing was obtained, whereas the unblended lubricating oil scored at 10,000 lbs./sq. in. pressure.

It will be seen, from the above examples, that the addition of an ester of a phosphorus acid to a mineral oil improves the lubricating value of such an oil to a marked extent, and imparts to the oil certain properties which render it suitable for use in the lubrication of surfaces engaging under extreme pressure.

While I have described my invention with reference to the lubrication of gears and bearings operating under heavy loads, I do not intend to limit myself thereto, but contemplate the use of my lubricant in operations such as the cutting and boring of metals, in which conditions of extreme pressure and temperature are normally encountered, and also in the lubrication of mechanisms operating under moderate pressures, as for example, the crankcase bearings and cylinder walls of internal combustion engines. Furthermore, my compounded oil may be utilized as a base in the preparation of thickened oils, i. e., greases by the addition thereto of soaps or other conventional thickening agents, whereby to obtain lubricants of desired viscosity. My compounded oil may also be blended with fatty oils, or the esters alone may be admixed with fatty oils, for the lubrication of mechanisms in which the presence of a fatty oil is desirable.

For brevity, in the appended claims, the term "viscous hydrocarbon oil" is to be understood to mean a hydrocarbon oil whose viscosity is at least as great as 50 seconds Saybolt universal at 100° F.

What I claim is:

1. A lubricant comprising a viscous hydrocarbon oil and a halogenated ester of a phosphorus acid.

2. A lubricant comprising a viscous hydrocarbon oil and less than substantially 10% of a halogenated ester of a phosphorus acid.

3. A lubricant comprising a viscous hydrocarbon oil and less than substantially 3% of a halogenated ester of a phosphorus acid.

4. A lubricant comprising a viscous hydrocarbon oil and less than substantially 1% of a halogenated ester of a phosphorus acid.

5. A lubricant comprising a viscous hydrocarbon oil containing chlorinated tri-cresyl phosphate.

6. A lubricant comprising a viscous hydrocarbon oil and less than substantially 10% of chlorinated tri-cresyl phosphate.

7. A lubricant comprising a viscous hydrocarbon oil and a halogen substituted aryl ester of a phosphorus acid.

8. A lubricating composition comprising a

BEST COPY AVAILABLE
from the original bound volume

major proportion of petroleum lubricating oil, and a minor proportion of an aryl phosphate, the latter in an amount sufficient to increase substantially the load bearing ability and decrease the wear between relatively moving metallic surfaces when said composition is used for the lubrication thereof.

9. The method of retarding wear, scuffing or seizing of relatively moving metallic surfaces, which comprises maintaining therebetween a thin lubricating film formed by the action thereupon of an aryl phosphate, which film provides effective lubrication at pressures and speeds at which mineral lubricating oils normally will not be retained between said surfaces, said film resulting from the interposition between said surfaces, of a petroleum lubricating oil containing as a thin film forming agent, a small amount of an aryl phosphate.

10. An extreme pressure lubricant consisting of mineral lubricating oil and tricresyl phosphate in an amount sufficient to increase the load carrying capacity of the mineral lubricating oil so as to prevent seizure and scoring of bearings lubricated thereby when operated under pressures of at least 10,000 lbs. per square inch.

11. A lubricant comprising a viscous hydrocarbon oil and a small amount of a halogenated aryl phosphate.

WILLIAM M. MALISOFF.

228

BEST COPY AVAILABLE

from the original bound volume

Marked

BRIEF FOR APPELLANT, THE LUBRI-ZOL CORPORATION.

United States Court of Appeals
For the District of Columbia

United States Court of Appeals
for the District of Columbia

FILED SEP 22 1947

Joseph W. Stewart
CLERK

THE LUBRI-ZOL CORPORATION,
Appellant,
v.
LAWRENCE C. KINGSLAND,
COMMISSIONER OF PATENTS,
Appellee.

APPEAL No. 9576.

18

APPEAL FROM FINAL JUDGMENT OF THE
DISTRICT COURT OF THE UNITED STATES
FOR THE DISTRICT OF COLUMBIA.

EDWIN R. HUTCHINSON,
1154 Munsey Building,
Washington 4, D. C.,
Counsel for Appellant.

Of Counsel:
OSCAR C. LIMBACH,
909 Leader Building,
Cleveland 14, Ohio.

THE GATES LEGAL PUBLISHING CO., CLEVELAND, OHIO—MAIN 5647

INDEX.

Statement 1

Appellant's Contentions 5

Propositions of Law Relied Upon 5

1. Questions of novelty are questions of fact 5

2. A prior patent to be an anticipation must bear within its four corners adequate directions for the practice of the patent sought to be anticipated 5

Argument 6

1. The Invention 6
   - Formula 1—*Phosphoric Acid* 7
   - Formula 2—*Mono Phenyl Phosphate* 7
   - Formula 3—*Diphenyl Phosphate* 8
   - Formula 4—*Triphenyl Phosphate* 8
   - Formula 5—*Monolorol Phosphate* 9
   - Formula 6—*Dilorol Phosphate* 9
   - Formula 7—*Dilorol Phenyl Phosphate* 10
   - Formula 8—*Cresyl Group* 11
   - Formula 9—*Monocresyl Phosphate* 11
   - Formula 10—*Tricresyl Phosphate* 12
   - Formula 11—*Dilorol Cresyl Phosphate* 12
   - Formula 12—*The Board of Appeals' Interpretation of Dilorol Cresyl Phosphate* 13

2. Are the Compositions Covered by the Claims in Issue Patentably Distinct from the Compositions of the Prior Art? 17

Conclusions 27

AUTHORITIES CITED.

Case.

Dewey & Almy Chemical Co. v. Mimex Co., 124 F. (2d) 986 ....5, 25-26

Texts.

Hackh's Standard Chemical Dictionary, "alkylation" 15

Robinson On Patents, Volume I, Section III, Articles 245 and 246, page 334 ....24, 25

Walker on Patents, Deller's Edition, Volume One, Section 61, page 294.............................5, 23

United States Court of Appeals For the District of Columbia

THE LUBRI-ZOL CORPORATION,
Appellant,
v.
LAWRENCE C. KINGSLAND,
COMMISSIONER OF PATENTS,
Appellee.

APPEAL NO. 9576.

APPEAL FROM FINAL JUDGMENT OF THE
DISTRICT COURT OF THE UNITED STATES
FOR THE DISTRICT OF COLUMBIA.

BRIEF FOR APPELLANT, THE LUBRI-ZOL CORPORATION.

STATEMENT.

Appellant, The Lubri-Zol Corporation, is the owner by assignment of a certain application for patent of Carl F. Prutton, Serial No. 197,732, filed March 23, 1938, for Improvements in "Lubricating Composition."

The lubricating compositions to which the application relates comprise a major proportion of mineral lubricating oil containing a minor amount of certain organic compounds of phosphorus. These phosphorus compounds are added to the oil for the purpose of improving the film strength or load-carrying capacity of the oil.

While the application was originally filed in 1938, the long pendency of the application has been due to a series of unusual circumstances in the Patent Office and for which the appellant was in no way responsible. On March 15, 1940, the Primary Examiner finally rejected the application, whereupon an appeal was had to the Board of Appeals, and that tribunal, in a decision dated June 30, 1941, (page 45*), reversed the Primary Examiner and allowed all of the claims then standing in the application.

After that decision by the Board of Appeals the application was returned to the Primary Examiner who then suspended action for some time to wait the issuance of certain patents then in the form of pending applications, and the Primary Examiner then, on October 12, 1942, (page 47), rejected the application on the newly issued patents to Malisoff and Downing. Further prosecution of the application was had which was concluded with a final rejection by the Primary Examiner and a second appeal to the Board of Appeals. The second decision by the Board of Appeals dated March 2, 1945, (page 59), affirmed the Primary Examiner in his rejection of all of the claims on the Downing and Malisoff patents. The Primary Examiner's Statement to the Board of Appeals in connection with this second appeal, in summarizing the grounds of final rejection, (page 52), stated that there was believed to be no invention in the use of the phosphorus compounds covered by the claims on appeal *in view of* tricresyl phosphate and certain mixed esters disclosed by Downing.

The Board of Appeals in its second decision based its conclusion on a new ground, namely that a compound "dilorol cresyl phosphate" and the "mixed tertiary-amylphenyl phosphates," disclosed by Downing, (page 60), were a direct anticipation of the claims on appeal in that its compounds were included within the scope of the definitions of phosphorus compounds contained in the appealed claims. This was the first time it was ever contended that dilorol cresyl phosphate was included within the scope of the claims in issue. Because of the fact that the Board of Appeals was technically in error as to the chemical structure of each of these compounds, appellant petitioned for reconsideration.

---

* All references to page number are to the pages of the joint appendix.

The Board of Appeals in its decision "On Request for Reconsideration," (page 62), reversed, in part, its previous stand and held that because of the showing made in the Petition for Reconsideration, the "*mixed* tertiary amylphenyl phosphates" were *not* included within the scope of the claims on appeal, but affirmed its previous decision as to the molecular structure of the "dilorol cresyl phosphate" disclosed by Downing and held that compound anticipatory of the claims on appeal.

It will be observed, therefore, that in the proceedings before the Board of Appeals during the second and third considerations by the Board of Appeals of this application, an issue arose which had not been previously formulated.

When the application went to the Board of Appeals the second time it was on the issue as to whether the compounds covered by the appealed claims were in their effect *patentably* different from the tricresyl phosphate disclosed by Downing. In its second decision the Board of Appeals gave little consideration to that issue but raised the new issue that dilorol cresyl phosphate disclosed by Downing was actually an example of the compounds claimed by the application and therefore a *direct* anticipation thereof.

Because of the nature of the proceedings as above outlined it became necessary for appellant to proceed under R. S. 4915 in order that additional evidence might be adduced on the question of the chemical identity of dilorol cresyl phosphate and the compounds covered by the claims in issue. An appeal to the Court of Customs and Patent Appeals would have been on the Patent Office record only, and that tribunal would not have had the benefit of any expert testimony on the technical question which thus first arose in the second and third decisions by the Patent Office Board of Appeals.

During the trial of the case before the district court considerable expert testimony was adduced on the technical question involved. However, the district court in his "Informal Memorandum," page 11, merely held that "I find the claims in suit unpatentable over Downing and Malisoff" without giving any technical reasons for such conclusion.

It is respectfully contended that the trial court did not fully understand the issue nor the technical aspects of the question, as evidenced by the fact that No. 5 of the "Findings of Fact," page 12, states that:

> "The diloral cresyl phosphate is an alkylated ester of a phosphorus acid of the type obtained by condensing a *halogenated* ester of a phosphorus acid with an unsaturated chain compound."

It requires only elementary chemical knowledge to determine that dilorol cresyl phosphate which contains *no* halogen cannot be produced and will *not* be produced by "condensing" or combining the molecule of a "*halogenated* ester of a phosphorus acid" with the molecule of "an unsaturated chain compound."

The present appeal is therefore concerned not so much with questions of law as it is with questions of fact.

APPELLANT'S CONTENTIONS.

The appellant, The Lubri-Zol Corporation, contends:

1. Neither the Downing nor Malisoff patents disclose any lubricating composition which is included within the scope of the lubricating compositions defined by the claims in issue.

2. The dilorol cresyl phosphate disclosed by Downing is not an alkylated aromatic ester of an acid of phosphorus of the type obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound as defined by claims 1, 4, and 7 nor has it the molecular structure of the chemical addition agent defined by the remaining claims in that the alkyl group does not contain the number of carbon atoms called for by the claims in issue.

3. The teachings of the Downing and Malisoff patents are directly away from the course followed by the inventor Prutton when he invented the compositions covered by the claims in issue.

4. While the compounds disclosed by Downing and Malisoff do have a certain degree of utility, the compositions covered by the appealed claims give such radically superior results that they are clearly patentable over any of the compositions disclosed by Downing or Malisoff.

PROPOSITIONS OF LAW RELIED UPON.

1. **Questions of novelty are questions of fact.**

Walker on Patents, Volume 1, Section 61, page 294.

2. **A prior patent to be an anticipation must bear within its four corners adequate directions for the practice of the patent sought to be anticipated.**

Dewey & Almy Chemical Co. v. Mimex Co., 124 F. (2d) 986.

ARGUMENT.

1. The Invention.

The twelve claims in issue, as set forth in the Complaint (pages 3 and 4), all relate to lubricating compositions which comprise a major proportion of a mineral lubricating oil to which has been added a minor amount of a certain chemical material for the purpose of, as stated in the specification of the application, improving the film strength of the oil and to reduce the tendency of the oil to oxidize and form sludge. The twelve claims in issue may be divided into the following three groups:

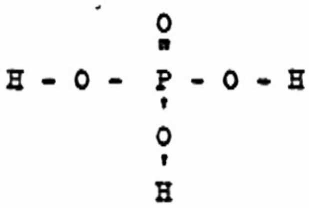
> (1) Claims Nos. 1, 4, and 7 which identify the chemical addition agent by the method of its manufacture or more specifically as the product obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound.
>
> (2) Claims Nos. 2, 5, 8, 10, 12, 13, and 14 which define the chemical addition agent in terms of its molecular structure irrespective of its mode of preparation. More specifically, such claims variously define the addition agent as an alkylated aromatic ester of a phosphorus acid containing an alkyl group having a specified minimum number of carbon atoms.
>
> (3) Claims Nos. 11 and 15 which define the addition agent in general as in the claims of group (2) excepting that such addition agent is further limited by the requirement, as specified in the claims, that it contain combined halogen in the molecule.

In order to simplify the necessary consideration of the claims and a comparison of the same with the disclosures of the Downing and Malisoff patents it may be well to review briefly the basic chemistry involved.

It will be observed that the compounds defined by the claims are in each instance an "ester." An ester is the organic compound produced when an organic group is substituted for the acidic hydrogen of an acid. Thus, as explained by the expert Dr. Lankelma, page 19, the molecular structure of phosphoric acid is as follows:

Formula 1.

Phosphoric Acid.



$$\begin{array}{ccccccccc} & & & & O & & & & \\ & & & & \| & & & & \\ H & - & O & - & P & - & O & - & H \\ & & & & | & & & & \\ & & & & O & & & & \\ & & & & | & & & & \\ & & & & H & & & & \end{array}$$

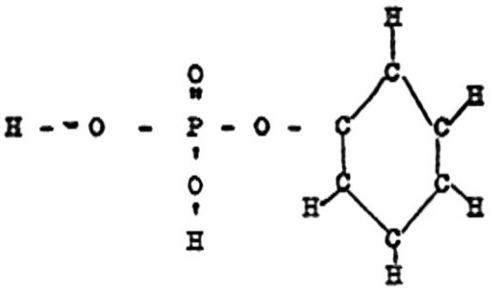
The above structural formula is Figure 1 of the chart, Exhibit 2 (page 65). In that formula P stands for phosphorus, O stands for oxygen, and H stands for hydrogen. The hydrogen atoms in this molecule are commonly referred to as "acid" hydrogen atoms in that their presence and location in the molecule is responsible for the *acidic* character of the compound. These acid hydrogens are quite readily replaceable. They may be replaced by a wide variety of other chemical groups. Various groups which may be substituted for these acid hydrogens are also shown diagrammatically in Plaintiff's Exhibit 2 (page 65).

When the well-known benzene ring, shown in Fig. 3 of Plaintiff's Exhibit 2, is used as a substituent group, as by the removal therefrom of one of its hydrogen atoms, it is known as the phenyl group, shown in Fig. 4 of Exhibit 2. When one phenyl group is substituted for one of the acid hydrogens of the phosphoric acid molecule of Formula 1 a compound of the following structure will be produced:

Formula 2.

Mono Phenyl Phosphate.

①



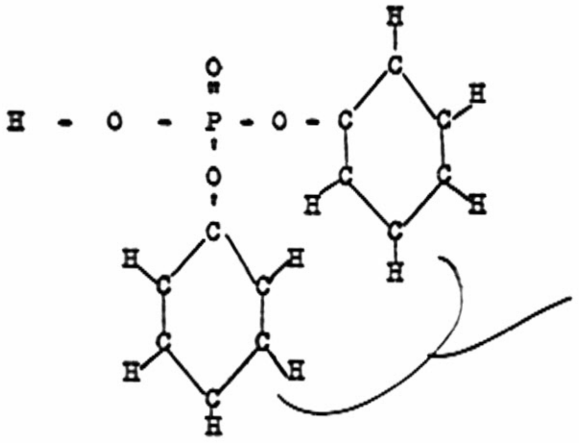
Phosphoric
+
Benzene

When *two* such phenyl groups are substituted for a corresponding number of acid hydrogens of the phosphoric acid molecule a compound having the following structure is produced:

Formula 3.

Diphenyl Phosphate.

②



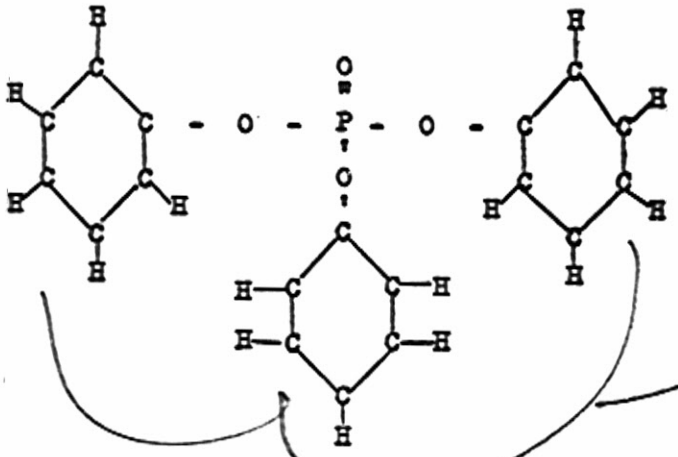
Benzene Ring

and when each of the three acid hydrogens are replaced by means of phenyl groups a compound having the following structure is produced:

Formula 4.

Triphenyl Phosphate.

③



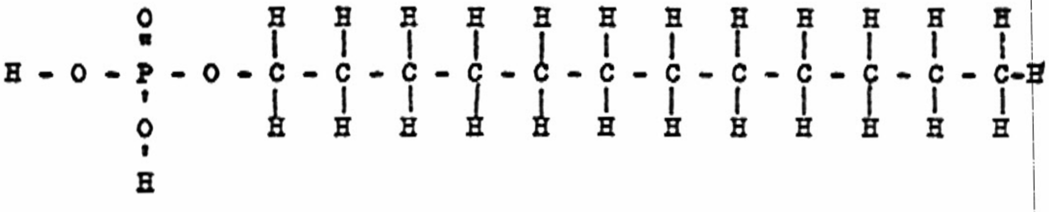
Ben Ri

Each of the three compounds shown in Formulas 2, 3, and 4 above, are called *esters.* The compounds of Formulas 2 and 3 are called *acid* esters because the molecule still contains an unsubstituted *acid* hydrogen.

Similarly, instead of using the phenyl group to replace these acid hydrogens of the acid molecule, one may use other groups. One such other organic group, for example, may be obtained from a chain compound instead of from a ring compound as was the phenyl group. In a chain compound the carbon atoms are hooked together in a chain rather than in a closed ring as in benzene. One such chain compound which has a straight chain of twelve carbon atoms is called the *lorol* group and when a lorol group is substituted in place of one of the hydrogens of the phosphoric acid molecule a compound having the following structure is produced:

Formula 5.

Monolorol Phosphate.

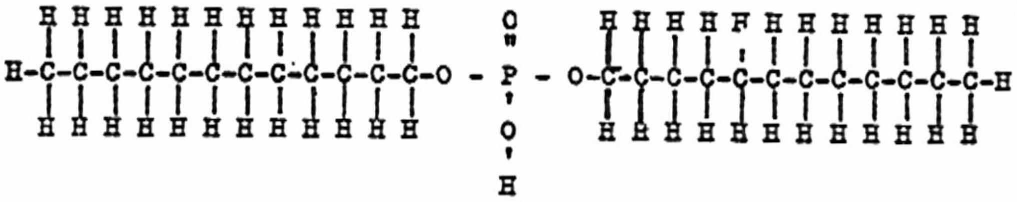


$$\begin{array}{ccccccccccccccccccccccccccccccccc}
&&&&O&&&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&\\
&&&&\|&&&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&\\
H&-&O&-&P&-&O&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&H\\
&&&&|&&&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&\\
&&&&O&&&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&\\
&&&&|\\
&&&&H
\end{array}$$

Now if two lorol groups are substituted for a corresponding number of hydrogen atoms of the phosphoric acid molecule a structure such as the following is produced:

Formula 6.

Dilorol Phosphate.

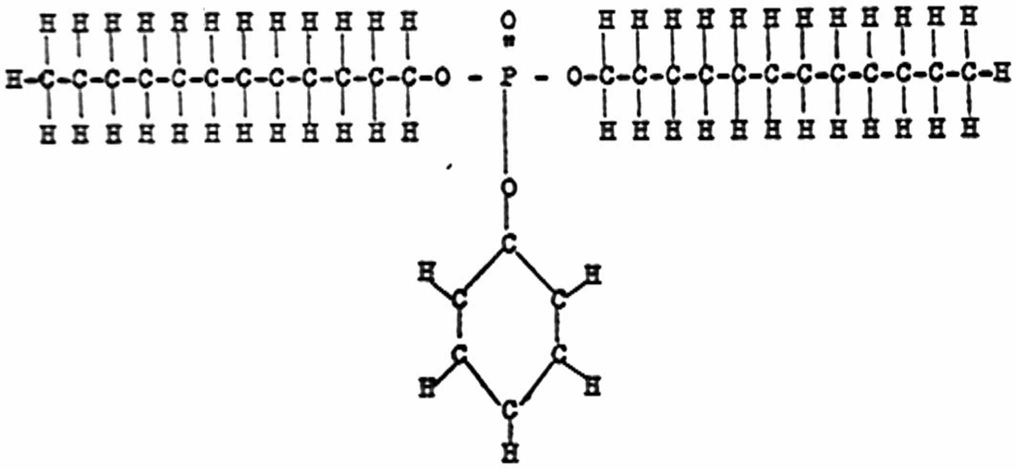


$$\begin{array}{ccccccccccccccccccccccccccccccccccccccccccccccccccccccccc}
&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&&&O&&&&H&&H&&H&&H&&F&&H&&H&&H&&H&&H&&H&&H&&\\
&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&&&\|&&&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&\\
H&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&O&-&P&-&O&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&C&-&H\\
&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&&&|&&&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&|&&\\
&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&&&O&&&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&H&&\\
&&&&&&&&&&&&&&&&&&&&&&&&&&&&|\\
&&&&&&&&&&&&&&&&&&&&&&&&&&&&H
\end{array}$$

It will be observed that the compound of Formula 6 still contains one replaceable acid hydrogen. Now if that acid hydrogen is replaced by a substituent group *other* than a lorol group, as for example, by a phenyl group, then a compound having the following structure is produced:

Formula 7.

Dilorol Phenyl Phosphate.



It will be noted that that compound is called "Dilorol Phenyl Phosphate," the term *dilorol* denoting the presence of *two* lorol groups and *phenyl* denoting the presence of *one* phenyl group.

One aryl + 2 chains

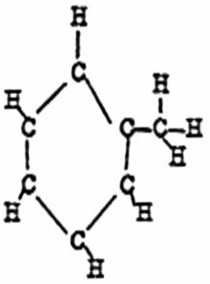
The compound of Formula 7 is a *mixed* ester because it has *different* kinds of substituent groups replacing the acid hydrogens of the acid nucleus.

In naming the various chain compounds a different name is used each time the length of the chain changes by one carbon atom and the names chosen are indicative of the number of carbon atoms present in the chain. Thus, for example, propane or the unsaturated three-carbon atom radicle derived therefrom contains three carbon atoms in the chain. When there is only one carbon atom present and its four valences are all satisfied by hydrogen we have methane, the principal constituent of natural gas commonly used for cooking purposes. When one of the acid hydrogens is removed from the methane molecule we have the methyl group shown in Fig. 6 of Plaintiff's Exhibit 2, (page 65).

It is also possible to combine various organic groups as, for example, the phenyl group of Fig. 4 of Plaintiff's Exhibit 2 and the methyl group of Fig. 6. When that is done we have a molecular structure as follows:

Formula 8.

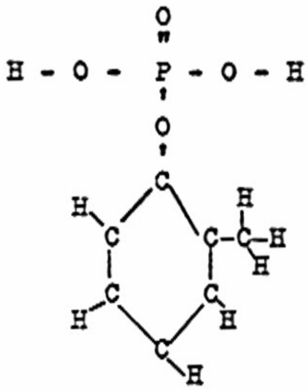
Cresyl Group.



Well established chemical nomenclature has chosen the word cresyl to identify the group of Formula 8, which is really a phenyl group having one methyl substituent thereon. When the cresyl group is used to replace one of the acid hydrogens of phosphoric acid, we produce a molecule having the following structure:

Formula 9.

Monocresyl Phosphate.

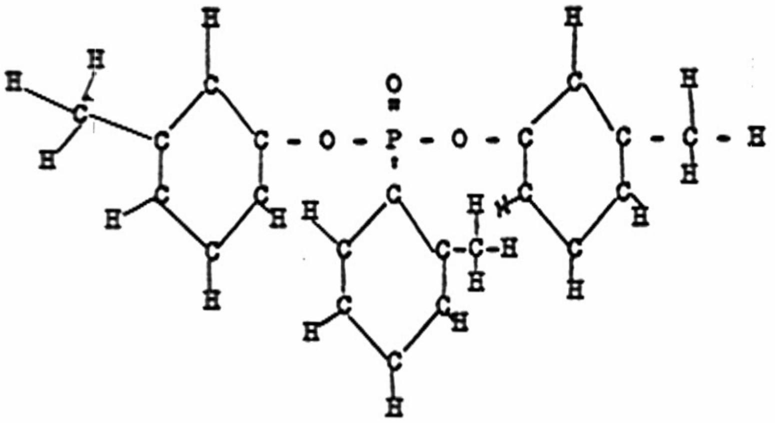


and when all three of the acid hydrogens of the phosphoric acid molecule have been replaced by a cresyl group of the structural formula of Fig. 8 then there is produced the following compound:

#11 is Dilorol Cresyl
#2 is Dilorol Cresyl'

Formula 10.

Tricresyl Phosphate.

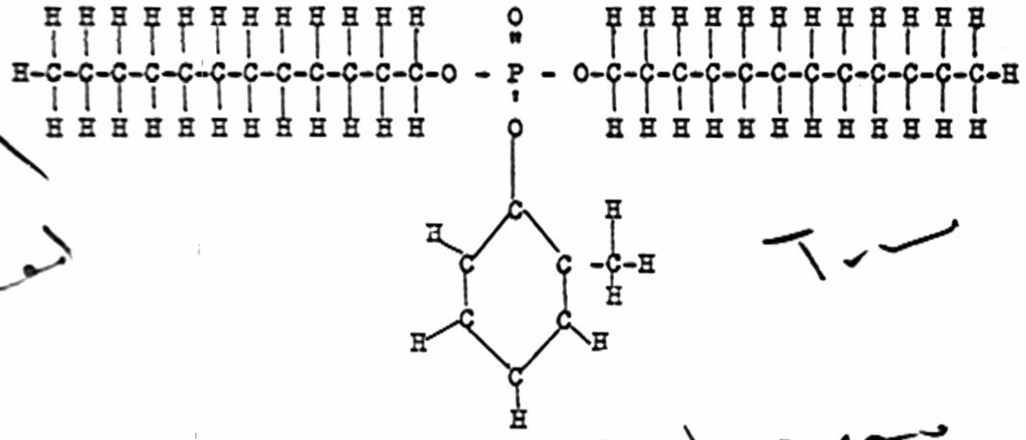


The compound thus produced is named "Tricresyl Phosphate" because there are three cresyl groups substituted for the corresponding number of hydrogen atoms of the original phosphoric acid molecule.

The compound of structural Formula 10, i.e., tricresyl phosphate, is not a mixed ester as was the compound of Formula 7 for example. Instead it is a non-mixed ester similar to the compound of structural Formula 4. If we now use a single cresyl group of structural Formula 8 and two lorol groups to replace the three acid hydrogens of the phosphoric acid molecule we produce the following structure:

Formula 11.

Dilorol Cresyl Phosphate.



Down the

This is the only patent to claim P.O. [illegible] [illegible]

True structure

All acid hydrogens replaced.

Is this same [illegible]
of hydrogens.

That compound is called "Dilorol Cresyl Phosphate" because it contains *two* lorol groups and *one* cresyl group. Dr. Lankelma thus constructed the molecule of this material (see Fig. 11, Plaintiff's Exhibit 4, page 71). Dilorol cresyl phosphate, the compound of structural Formula 11, was the compound disclosed by Downing.

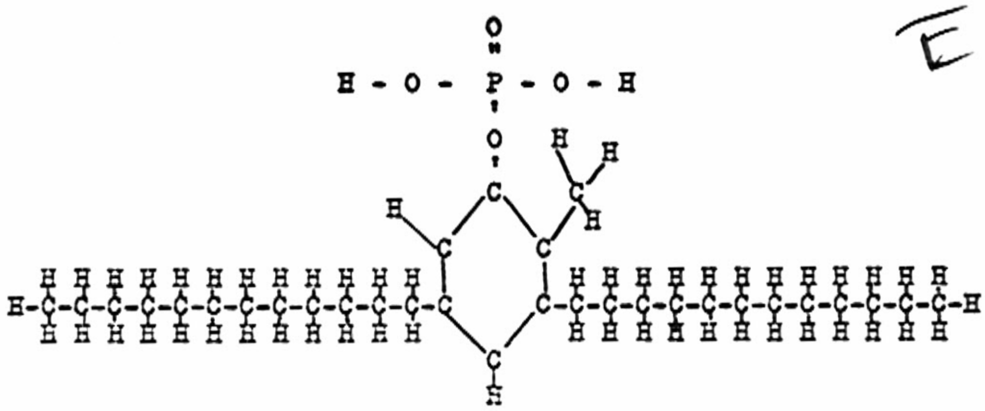
The Patent Office Board of Appeals in its "Request For Reconsideration," (page 62), in referring to dilorol cresyl phosphate, said:

> "This should mean a compound where the two lorol radicles of fifteen* carbon chains each are attached to the benzene ring of the cresyl radicle rather than to the phosphoric acid radicle."

In other words, the Board of Appeals considered that the structural formula of dilorol cresyl phosphate was as follows:

Formula 12.

The Board of Appeals' Interpretation of Dilorol Cresyl Phosphate



Error?

Only one acid hydrogen replaced, at bottom.

The compound of structural Formula 11 is a *mixed* ester and the compound of Formula 12 is *not* a mixed ester. We now turn to the Downing patent to determine whether his disclosure sheds any light on this question.

---

* While the Board of Appeals referred to the lorol group as containing "15" carbon atoms the record is clear that it actually contains only 12 carbon atoms.

There is of record as Plaintiff's Exhibit 3, extracts from the file of the Downing patent. During the prosecution of the application which resulted in the Downing patent the attorneys for Downing proposed a claim 61 in which the addition agent was identified as "a mixed ester of an acid of phosphorus." They also proposed claim 82 page (68) in which the addition agent was identified as dilorol cresyl phosphate. Then in the concluding paragraph of that Exhibit (page 69) the attorneys for Downing stated "Claim 61 is generic to each of claims 79-82," clearly establishing that Downing considered the dilorol cresyl phosphate disclosed by him as a "mixed" ester, in which case it would need to have the structure of structural Formula 11 and could not have the structure of structural Formula 12.

The Patent Office Board of Appeals in its decision "On Request for Reconsideration," (page 62), reconsidered its previous holding that the mixed esters were anticipatory and reversed its position on that point and held that the mixed esters were not anticipatory of the present invention. The Patent Office Board of Appeals then went on to state that dilorol cresyl phosphate is not a mixed ester and accordingly held it anticipatory. Had the Patent Office Board of Appeals been fully advised of the true structural formula of dilorol cresyl phosphate they would have, if consistent with their other holding, held that dilorol cresyl phosphate is not anticipatory.

No evidence has been adduced in support of the contention that the dilorol cresyl phosphate of Downing has the structure of Formula 12.

During the trial of this case in the district court, the solicitor for the appellee contended that even if dilorol cresyl phosphate has a structure of structural Formula 11 that compound is still an alkylated aromatic ester of an acid of phosphorus, as called for by the claims in issue and, as a matter of fact, the "Findings of Fact" and "Conclusions of Law" promulgated by the solicitor for the appellee and adopted by the trial court after the issuance of the trial court's "Informal Menorandum" contains as point six a holding that dilorol cresyl phosphate is an alkylated aromatic ester of a phosphorus acid containing an alkyl group having at least two carbon atoms. That holding thus adopted by the trial court was clearly in error for the following reasons:

The standard chemical dictionary is *Hackh.* That dictionary defines the term "alkylation" as follows:

> "The substitution of an aliphatic hydrocarbon radicle for a hydrogen atom in a cyclic compound; as, the introduction of a side chain to an aromatic compound."

Thus, alkylation is the union of a cyclic compound and an aliphatic hydrocarbon, i.e., the union of dissimilar molecules. The union of similar molecules, if they are both cyclic, is commonly termed "arylation," and, if they are both aliphatic, is commonly termed either "condensation" or "polymerization."

It is important to note that the Patent Office in its classification of patents pertaining to organic chemical compounds likewise has followed the well-accepted definition of alkylation as meaning "the introduction of a side chain to an aromatic compound." Class 260 of the classification of patents in the Patent Office is concerned with the chemistry of carbon compounds, and sub-class 671 therein is the sub-class having to do with alkylation. In Bulletin No. 85 of the United States Patent Office wherein are given the definitions of the various sub-classes of class 260, class 671 is defined as patents on processes "which are directed to the *introduction of an acyclic hydrocarbon side chain into a benzene nucleus.*"

It cannot be contended that the true structure of dilorol cresyl phosphate as shown by Formula 11 is an alkylated aromatic ester of an acid of phosphorus if one is to have any degree of respect for common chemical nomenclature.

The term "alkylation" can never be interpreted to mean the introduction of an aliphatic hydrocarbon substituent into an *inorganic* group which would need to be the case where the lorol groups are attached to the phosphate group as in Formula 11. The latter process (that is where an organic group replaces an acid hydrogen) is referred to in standard chemical nomenclature as "esterification." "Alkylation" and "esterification" are entirely independent, separate, and distinct processes and neither term is inclusive of any part of the other.

Every single example given in the specification of the patent application under consideration is of the type in which the alkyl substituent is on a cyclic group, that is, a true alkylated aromatic compound. Even without the standard chemical nomenclature referred to above, the claims in the application under consideration would necessarily be interpreted to mean that the alkyl group is on the cyclic nucleus and it was so interpreted by the Board of Appeals when it sought to spell out an anticipation by contending that in the molecular structure of dilorol cresyl phosphate the lorol groups are attached to the cyclic nucleus.

All of the foregoing discussion has been concerned with those claims wherein the compounds used as addition agents are identified by their molecular structure. Those claims are Nos. 2, 5, 8, 10, 12, 13, and 14. Each of those claims identifies the addition agent as an alkylated aromatic ester of a phosphorus acid in which the alkyl group has at least two and preferably more than four carbon atoms. Tricresyl phosphate is distinguished over by those claims in the number of carbon atoms which are required to be present in the alkyl substituent on the benzene ring. As to whether that difference in number of carbon atoms leads to a patentable distinction we leave for discussion at a later point in this brief.

Claims 1, 4, and 7 identify the addition agent by the method of its manufacture and identify it more specifically as the product obtained by condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound. This language was selected purposely to distinguish over tricresyl phosphate, and Dr. Lankelma has testified at length that *tricresyl phosphate cannot be made by the process of condensing an aromatic ester of a phosphorus acid with an unsaturated chain compound* (page 26, line 9, *et seq.*).

The only remaining claims, Nos. 11 and 15, define the addition agent in terms of its chemical structure except that such addition agent is further limited by the requirement that it contain combined halogen in the molecule. The previously considered claims were shown to distinguish chemically over tricresyl phosphate. As anticipatory of the claims 11 and 15 the Patent Office has relied on the disclosure of Malisoff which mentions halogenated tricresyl phosphate. Malisoff is therefore no closer an anticipation of claims 11 and 15 than is Downing an anticipation of the other claims.

By means of the foregoing consideration of basic chemical principles and accepted chemical nomenclature we have shown conclusively that the definitions of the addition agents as employed in the claims in issue do not include within their scope any of the compounds disclosed by the prior art. We come now therefore to a consideration of the other principal question involved in this appeal, i.e., whether the compounds covered by the claims in issue are patentably distinct from the compounds disclosed by Downing and Malisoff.

2. Are the Compositions Covered by the Claims in Issue Patentably Distinct from the Compositions of the Prior Art?

The phosphorus compounds characterizing the lubricating compositions of the claims in issue are represented by Fig. 12 of Plaintiff's Exhibit 4, (page 71). The two compounds of the prior art are shown by Fig. 11 of Plaintiff's

Exhibit 4, (page 71), which gives the structural formula for dilorol cresyl phosphate and the structural Formula No. 10 given in this brief which shows the structure of tricresyl phosphate. Comparing those three structural formulae it will be noted that dilorol cresyl phosphate of Fig. 11 of Plaintiff's Exhibit 4, is very radically different in structure from the type of compound with which the claims in issue are concerned, as represented by Fig. 12.

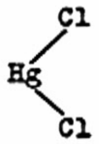
Comparison

A comparison of the structural formula of tributyl phenyl phosphate of Fig. 12, Plaintiff's Exhibit 4, with the structural formula of tricresyl phosphate given by structural Formula 10 in this brief shows a greater apparent similarity. During the proceedings before the trial court the solicitor for the Patent Office contended that tributyl phenyl phosphate and tricresyl phosphate are members of the same homologous series, differing from each other in the number of carbon atoms in the alkyl or aliphatic substituent groups which are attached to the phenyl or ring groups. Dr. Lankelma was questioned on this point during his testimony and he stated that the greatest difference between adjacent members of a homologous series is "between the first member and the second, that is, between the single carbon member and the following member which contains two carbons." (page 27, line 19). In other words, tricresyl phosphate may be considered the first member of a homologous series in that the alkyl substituent contains one carbon atom. The butyl compound of Fig. 12 of Plaintiff's Exhibit 4 is the fourth member of the series in that it contains four carbon atoms in the aliphatic substituent. A skilled chemist such as Dr. Lankelma would therefore expect there to be a great difference between these menbers of the series because tricresyl phosphate is the first member and the butyl compound is the fourth.

Dr. Lankelma then continued (page 27) by stating that methyl alcohol, which is the alcohol containing one carbon atom in the chain is wood alcohol and is a poison whereas ethyl alcohol which contains two carbon atoms in the chain is ordinary grain alcohol and is used for beverage purposes.

Dr. Lankelma might also have compared two other well-known chemicals, namely the following compound:

Corrosive Sublimate
(a deadly poison)



with

Calomel
(a medicine taken internally)

$Hg—Cl$

In considering the structural formulae which we have given in our brief it must be kept in mind therefore that changes which on paper appear to be merely slight changes, when translated to the finished chemical compound may be very radical changes indeed.

During the course of the proceedings before the trial court the solicitor for the appellee contended that from the teachings in the Downing patent one would be led to try the compositions covered by the claims in issue. The data contained in the Downing specification lead to quite an opposite conclusion. Downing lists seventeen specific examples of his invention. In these examples various amounts of various addition agents were employed. Two factors were determined in evaluating each example. Those factors were the load carrying capacity and the torque.

In the following table we have summarized the results secured by Downing from his tests on these seventeen examples:

Example No.	*Addition Agent*	*%*	*Load Carried*	*Torque*
1	Dilauryl phosphate	2.0	55,000	2.2
2	Dicetyl phosphate	1.0	55,000	2.0
3	Tricresyl phosphate	5.0	25,000	3.5
4	Dicyclohexyl phosphate	1.0	20,000	1.4
5	Dioleyl phosphate	1.0	45,000	2.0
6	Dilauryl phosphate	1.0	52,000	2.2
7	Di- and Tri-lorol phosphites	1.0	51,000	2.3
8	Triphenyl phosphite	1.0	48,000	3.2
9	Trinaphthyl phosphate	1.0	21,000	2.2
10	Dilorol phosphate	1.0	45,000	2.2
11	Mixed dilorol phosphates	1.0	32,000	2.1
12	Lorol and ocenol phosphates	?	45,000	2.1
13	Lecithin	5.0	52,000	3.0
14	Dilorol phosphate	0.5	60,000	2.4
15	Dilorol phosphate (emulsified)	1.0	32,000	1.9
16	Salt of dilorol phosphate	1.0	35,000	2.4
17	Dilauryl dithiophosphate	1.0	55,000	2.4

The one compound in the foregoing list which the Patent Office has contended would lead a worker in the art in the direction of the compounds covered by the claims in issue is tricresyl phosphate.

Tricresyl phosphate gave the poorest results of any compound tried by Downing.

Dilorol phosphate, which is a straight aliphatic ester and does not contain any of the aromatic structures which characterize the compounds covered by the claims in issue, imparted to the oil a load carrying capacity of 60,000 pounds per square inch when used in a concentration of only one-half per cent. Tricresyl phosphate when used in a concentration ten times as great, i.e., 5%, imparted a load carrying capacity of only 25,000 pounds per square inch. Moreover, the torque, that is the friction, was 3.5 when using tricresyl phosphate, which was the highest torque observed in any of the samples tested. The teaching of Downing, therefore, which is most clearly expressed in the data given in connection with his seventeen specific examples, would lead in the direction of the straight aliphatic esters and *away* from the aromatic esters represented by tricresyl phosphate.

Downing was not the first to use a phosphorus compound in a lubricant. The applicant in the present case does not claim to be the first to use a phosphorus compound in a lubricant. Downing, working in this broad field of phosphorus compounds, found that a certain type, namely the aliphatic esters, were particularly suitable especially for extreme pressure properties. The inventor in the present case worked in an entirely different direction. He found that the alkylated aromatic esters which have a specified minimum number of carbon atoms in the alkyl substituent connected to the aromatic group unexpectedly gave "radically superior" results.

Because of the very poor results which Downing secured using tricresyl phosphate, it cannot be said that his patent "pointed the way" along the path which was followed by the present inventor.

That the compositions covered by the claims in issue are radically superior to the compositions of the prior art, as represented by tricresyl phosphate, is clearly demonstrated by the data of record in the affidavit of Albert K. Smith, (page 56). Dr. Smith was called as a witness during the proceedings before the trial court and he explained at length the data thus tabulated on page 56 as well as the reasons for the selection of the particular materials which were tested and the amounts of each which were selected for use.

The Patent Office, in its consideration of Dr. Smith's affidavit, criticized it in the following particulars:

> (1) The tests did not use the same amounts of each material.
>
> (2) The tests did not show any improvement as to oxidation inhibition.
>
> (3) The tests did not show any improvement in lessening of sludging.

Dr. Smith, in his testimony, beginning on page 34 and particularly with the second paragraph on page 35, explains in considerable detail wherein the Patent Office Board of Appeals was in error in its interpretation of his affidavit.

First with regard to the difference in the amount of the two materials used, this is explained by Dr. Smith on a scientific basis at the top of page 35 where he points out that the differences in amount were carefully calculated in order that the ultimate lubricant being tested would have present therein *equal amounts of the active components* of the separate compounds. If equal weights of the two compounds had been used, the results of the test would have been misleading in that one sample of oil would have contained therein a considerably greater amount of phosphorus than the other.

With regard to the data showing improvement as to oxidation inhibition, this Dr. Smith answered in his last answer on page 35 and also in the first answer on page 36 where he pointed out that the Conradson carbon test and the acid number determinations are standard tests which are relied on by those skilled in the art to determine information which will indicate the degree of oxidation of the oil. Dr. Smith also pointed out that the "naphtha insolubles" and "chloroform solubles" are further indications of the degree of oxidation of the oil.

Now with regard to the data showing improvement in sludging tendencies of the oils under test, Dr. Smith pointed out in his last answer on direct examination at the top of page 36 that the data previously identified in connection with oxidation improvement and notably the "naphtha insolubles" are also clearly indicative of the sludging tendency of the oil.

Since the addition agents used by Downing and Malisoff have been shown to be chemically different from those called for by the claims in issue the principal issue in this appeal is, therefore, whether the compounds covered by the claims in issue are the "equivalent" of tricresyl phosphate disclosed by Downing. In the first place, we wish to point out that under the law, novelty is a question of fact and questions of novelty belong to the province of evidence and not to that of construction. "*Walker on Patents,*" Deller's Edition, Volume One, Section 61, page 294, states the following:

> "Questions of novelty are questions of fact. (*Battin v. Taggert,* 17 How. (58 U. S.) 74, 15 L. Ed. 37 (1854); *Turrill v. Railroad Co.*, 1 Wall. (68 U. S.) 491, 17 L. Ed. 668 (1863). This point is very obvious, except in cases where the prior thing is a patent or printed publication. In those cases it may be supposed that questions of novelty are questions of law arising on the construction of documents. The point has, however, been settled by the Supreme Court, in a case involving the consideration of a prior patent, and bearing with equal logical force upon a prior printed publication. (*Bishoff v. Wethered,* 9 Wall. (76 U. S.) 812, 19 L. Ed. 829 (1869).) In that case it was held that the question whether the novelty of a patent is negatived by a prior patent, depends not upon the construction of the latter, but depends rather upon the outward embodiment of the terms contained in the latter document; and that such outward embodiment is to be properly sought, like the explanation of latent ambiguities arising from the description of external things, by evidence *in pais.* The court accordingly indorsed the proposition that such questions belong to the province of evidence, and not to that of construction; and said that even where no testimony is required to explain the terms of art or the description contained in the respective documents, the question is still to be treated as a question of fact."

Thus, instead of determining the question of novelty by hypothetical reasoning from the mere written word of the prior art patent relied upon as a reference, novelty can only be determined by evidence or actual facts. Such actual facts have been adduced and they are summarized in Dr.

Smith's statement which occurs at line 26 on page 36 where he stated that the compound tested by him which falls within the claims here in issue was "radically superior" to the compound representative of the Downing patent.

Unquestionably the most erudite and exhaustive treatise on the subject of patents is "*Robinson On Patents.*" In that work, in Volume I, Section III, Articles 245 and 246, beginning on page 334, occurs the following fundamental statement applicable to the present situation:

> "245. Inventions which perform the same functions may differ not only in shape, size, capacity, proportions, arrangement, and materials, but also in the individual character of the parts or elements of which they are composed. When such diversity results in the expression of a different idea of means, the diversity is one of substance, and each of the inventions is distinct from and independent of the other. When the idea of means in both inventions is essentially the same, the variation either indicates a different development of this idea, by which the later invention becomes an improvement on the earlier, or is a simple alteration in the form of its embodiment. The rules which govern these diversities are based on the familiar principles already stated, and taken together, constitute the 'Doctrine of Equivalents.'
>
> "246. The term 'equivalent' is used in Patent Law in two different senses, and in relation to two different subjects. In one sense it denotes the correspondence between agencies which not only perform the same functions, but are in themselves the same operative means. In this sense it is synonymous with 'identical'; and can be properly employed only in reference to an invention as a whole. In its second and more technical sense it signifies the interchangeability of agencies which are known in the arts to be capable of serving the same purpose as integral parts of some particular invention. In this sense it is applicable to the elements or ingredients by whose union in one art or instrument the inventor has embodied his idea of means. It does not indicate identity either in essential character or

> individual function, but merely the ability to produce the same effects when brought into connection with the other elements in the invention. It is in this sense that the word is used in speaking of the 'Doctrine of Equivalents.' "

As stated by *Robinson*, the real issue to be determined is whether the compounds covered by the claims here in issue are the "equivalent" of the compounds disclosed by Downing. Your Honors are fully familiar with the axiom:

> "That which would infringe if later would anticipate if earlier."

Not a single material disclosed by Downing comes within the scope of the claims here in issue. As so aptly stated by *Robinson* in the previously given quotation, only the full "equivalents" of a prior art patent may be considered to be within the teaching of that patent. *Robinson* defines "equivalents" as those "agencies which not only preform the same functions, but are in themselves the same operative means." The test data submitted by Dr. Smith show conclusively that the materials covered by the claims in issue give results which are "radically superior" to those of the compounds disclosed by Downing.

During the proceedings before the trial court the solicitor for the appellee contended that the invention defined by the claims in issue was nothing more than the result of a normal research program. It is believed that we have clearly shown that the use of tricresyl phosphate as disclosed in the Downing patent does not point the way in the direction of the present invention since the results secured by the use of that material by Downing was the poorest of all of the materials tried by him. There is always a strong temptation to interpret a prior art patent in the light of after-acquired knowledge.

Judge Learned Hand in the case of *Dewey & Almy Chemical Co. v. Mimex Co.*, 124 F. (2d) 986, said, beginning on page 989 of that decision:

> "No doctrine of the patent law is better established than that a prior patent or other publication to be an anticipation must bear within its four corners adequate directions for the practice of the patent invalidated. If the earlier disclosure offers no more than a starting point for further experiments, if its teaching will sometimes succeed and sometimes fail, if it does not inform the art without more how to practice the new invention, it has not correspondingly enriched the store of common knowledge, and it is not an anticipation."

In that case, Judge Hand had before him one of those developments which the opponents of the patent claimed it to be only the result of a normal research program. In holding the claims of the patent valid, Judge Hand first pointed out that it was true that the invention under consideration did not constitute a great pioneer invention. In the concluding portion of his opinion, Judge Hand made the following statement which is applicable to the facts before us here:

> "It would indeed be absurd to rank the invention as a great pioneer such as come only at rare intervals and are the work of genius. Indeed, it is precisely those which probably need no patents to call them forth; the stimulus of profit has little or no part in their production. The patent law is aimed at animating a lower order of imagination and skill than that; more, it is true, that the ordinary rub of competition automatically brings out from competent workmen in the art, but not the superlative skills—at least that has been its uniform avowed purpose. Perhaps the system is outworn, but while it stands, it stands clothed with its history like any other statute, and it seems to us that not to recognize so substantial an achievment as this which has resulted in the improved preservation of foods and other perishable goods, would deny recognition where recognition most is helpful."

CONCLUSIONS.

The record clearly shows:

1. No composition identified by any prior art patent is included within the scope of any of the claims in issue; and

2. The closest approach in the prior art to the compositions covered by the claims in issue is tricresyl phosphate. The Downing patent which gave comparative data on the effectiveness of tricresyl phosphate and a large number of other phosphorus compounds tried by Downing showed that of those materials tried by Downing tricresyl phosphate gave the poorest results. His teaching, therefore, leads away from the compositions covered by the claims in issue rather than in the direction of such compositions.

Wherefore we respectfully pray that the holding by the trial court be reversed and the claims in issue allowed to appellant.

Edwin R. Hutchinson,
Counsel for Appellant,
1154 Munsey Building,
Washington 4, D. C.

Of Counsel:

Oscar C. Limbach,
909 Leader Bldg.,
Cleveland 14, Ohio.

United States Court of Appeals
for the District of Columbia

FILED OCT 8 - 1947

Joseph W. Stewart
CLERK

BRIEF FOR APPELLEE

United States Court of Appeals

DISTRICT OF COLUMBIA

Appeal No. 9576

THE LUBRI-ZOL CORPORATION, APPELLANT

v.

LAWRENCE C. KINGSLAND, COMMISSIONER OF PATENTS,
APPELLEE

W. W. COCHRAN,
Solicitor, U. S. Patent Office,
Attorney for Appellee.

E. L. REYNOLDS,
Of Counsel.

INDEX

Page

Introduction 1

Appellant's application 1

References relied on 2

Summary of argument 2

Argument 2

Diloral cresyl phosphate 4

Tricresyl phosphate 6

Conclusion 11

Authorities cited:

- Chamberlain's Organic Chemistry, P. Blakiston's Sons, Philadelphia, 1934, Third Edition, page 21 8
- Gauerke, 86 F. 2d 330 11
- Hass et al, 141 F. 2d 122 9
- Minnesota Mining and Mfg. Co. v. Coe, 79 App. D. C. 217, 99 F. 2d 986 11
- Naamlooze Venootschafs v. Coe, 76 U. S. App. D. C. 313, 132 F. 2d 573 11
- Webster's New International Dictionary 7
- Wertheim's Textbook of Organic Chemistry, published by The Blakiston Co. of Philadelphia, Copyright 1939, page 32 7

United States Court of Appeals

DISTRICT OF COLUMBIA

Appeal No. 9576

THE LUBRI-ZOL CORPORATION, APPELLANT
v.
LAWRENCE C. KINGSLAND, COMMISSIONER OF PATENTS,
APPELLEE

APPEAL FROM THE JUDGMENT OF THE DISTRICT COURT OF THE UNITED STATES FOR THE DISTRICT OF COLUMBIA

BRIEF FOR THE COMMISSIONER OF PATENTS

INTRODUCTION

This is an appeal from the judgment (13)[1] of the District Court of the United States for the District of Columbia dismissing the complaint (2) in an action under R. S. 4915 (U. S. C., title 35, sec. 63) in which the appellant sought to have the Court authorize the issuance of a patent containing claims 1, 2, 4, 5, 7, 8, and 10 to 15, inclusive, of his application No. 197,732 for patent on a lubricating composition. These claims appear in the complaint (3, 4).

APPELLANT'S APPLICATION

The application here involved discloses a lubricant comprising mineral oil and a small amount of alkylated aromatic ester of an acid phosphorus. Among the esters suggested are tri-tertiary amyl phenyl phosphate and tri-butyl phenyl phosphate.

---

[1] The numbers in parentheses refer to pages of the appendix.

REFERENCES RELIED ON

The patent to Downing et al., 2,285,853, discloses a lubricant comprising a mineral oil and a small amount of alkylated aromatic ester of an acid of phosphorus. Among the esters suggested are tricresyl phosphate and di-lorol cresyl phosphate.

The patent to Malisoff, No. 2,289,509, discloses a lubricant comprising a mineral oil and a small amount of halogenated tri-cresyl phosphate.

SUMMARY OF ARGUMENT

1. The Downing patent discloses the use of either di-loral cresyl phosphate or tricresyl phosphate as an addition agent for mineral lubricating oil.

2. The appealed claims define nothing patentable over Downing's use of diloral cresyl phosphate.

3. The appealed claims define nothing patentable over Downing's use of tricresyl phosphate.

ARGUMENT

The appellant seeks to have the Court authorize the allowance of twelve claims of the application of Prutton, No. 197,732, for patent on a lubricating composition. The composition claimed comprises a mineral lubricating oil and a small amount of a material which, in claim 2, for example, is defined as "an alkylated aromatic ester of a phosporous acid containing an alkyl group having at least two carbon atoms." In claims 11 and 15 the product is said to be halogenated but, since halogenation of compounds of the type claimed here is conventional, as shown, for example, in the Malisoff patent, and since it does not appear to be alleged that halogenation produces any unexpected result in the particular compounds claimed here, it is thought that this feature need not be further considered. If the compounds are not patentable alone, halogenation cannot make them so and, accordingly, claims 11 and 15 stand or fall with the other claims.

As was fully explained in the testimony, the present compounds are esters which may be regarded as derived from phosphorous acid or phosphoric acid by replacing some or all of the hydrogen of the acid by one or more alkylated aromatic groups. Each of the present claims is limited to a compound in which the alkyl group contains at least two carbon atoms. A typical compound given in the application is tri-butyl phenyl phosphate. This was the compound chosen by Dr. Smith to demonstrate the alleged superiority of the compounds claimed here over those of the prior art.

The reference principally relied on here is the Downing patent and, specifically, two of the compounds disclosed therein, namely, tricresyl phosphate and diloral cresyl phosphate. It is submitted that the appellant's claims define no invention over tricresyl phosphate and that they are literally anticipated by diloral cresyl phosphate. These grounds will be considered separately. They are independent of each other and, if either is found to be proper, the complaint was properly dismissed.

DILORAL CRESYL PHOSPHATE

Diloral cresyl phosphate admittedly contains an alkyl group having enough carbon atoms to satisfy the requirements of the claims here involved. The Board of Appeals took the position that this group was attached to the phenyl ring of the compound, whereas the appellant contends that it is attached to the oxygen of the acid. This involves a rather technical point, which need not be determined here, since none of the claims specifies that the alkyl group is attached to the ring. In fact, Dr. Lankelma testified on cross-examination (29) that diloral cresyl phosphate, even as he construed that term, is "an alkylated aromatic ester of a phosphorous acid containing at least two carbon atoms", which is the exact language of claim 2 here involved. The witness contended that diloral cresyl phosphate is an acid ester, but this is immaterial, since the claims do not exclude acid esters. In view of this testimony of the appellant's witness it seems unnecessary to consider claims such as claim 2 in great detail. It may be noted, however, that Downing adds a small amount of his diloral cresyl phosphate to mineral lubricating oil, that diloral cresyl phosphate is an aromatic ester of a phosphorous acid, since the cresyl contains a phenyl ring, that the ester is alkylated by the loral group and that this group contains twelve carbon atoms. Claim 2, therefore, is fully satisfied. For the same reasons claims 5, 8, and 10 are satisfied. Claims 13 and 14 are similar to claim 2 but are limited to a phosphite, while claim 2 is broad enough to include phosphites and phosphates. There is clearly no patentable distinction between these compounds, and claims 13 and 14 are also substantially met by the diloral cresyl phosphate of Downing. Claims 11 and 15 add the statement that the compound is halogenated but, as above pointed out, halogenation would be obvious, especially in view of Malisoff.

Claims 1, 4, and 7 state that the compound is "of the type obtained by condensing an aromatic ester of a phosphorous acid with an unsaturated chain compound." This is clearly too indefinite, in itself, to warrant allowance of a claim. It is not stated that the compound was actually formed in the way stated, and the appellant's application, page 4, clearly states that tricresyl phosphite, which cannot be formed in this way, is "the type of addition agent contemplated by this invention". Evidently therefore, claims 1, 4, and 7 can only mean that the compound is similar to compounds which can be made in the manner recited. This is true of diloral cresyl phosphate which is quite similar to compounds made in the specified way, since it is, as they are, an alkylated aromatic ester of a phosphorous acid, containing more than one carbon atom in the alkyl group. Claims 1, 4, and 7, therefore, are unpatentable for the same reasons as claim 2.

Claim 12 is limited to tri-tertiary amyl phenyl phosphate. This is merely one compound of the group defined in the other claims. It has not been shown that it is superior in any way to the other compounds of the group and it is therefore evident that, if the other claims are unpatentable, there is no basis for allowing claim 12.

It is submitted that as the claims are worded, they are fully anticipated in all material respects by Downing's use of diloral cresyl phosphate. If it was the appellant's intention to limit the claims to compounds in which the alkyl group is attached to the phenyl ring, he should have done so by express language. Having worded his claims broadly, he should not be allowed to urge a narrow interpretation of them in order to avoid the prior art. Further, there is no evidence to show that it is material whether the alkyl group is attached to the phenyl ring or to some other part of the compound. Accordingly, even if it be assumed that the claims are not literally satisfied by diloral cresyl phosphate, there is no showing of a *patentable* distinction.

TRICRESYL PHOSPHATE

Tricresyl phosphate may also be designated as trimethyl phenyl phosphate. It is identical with tributyl phenyl phosphate, which is typical of the appellant's compounds, except that in tricresyl phosphate the alkyl group attached to the phenyl ring is a methyl group containing one carbon atom and three hydrogen atoms, whereas in tri-butyl phenyl phosphate, the corresponding alkyl group is a butyl group containing four carbon and nine hydrogen atoms. As was brought out in the testimony, these butyl and methyl compounds are homologs. It is unnecessary to consider what relationship makes compounds homologous, since it is not denied by the appellant that his tri-butyl phenyl phosphate is a homolog of the tri-cresyl phosphate of Downing. The issue therefore is simply whether, when it was known in the art that one member of a homologous series could be added to a mineral lubricating oil and would improve the properties thereof, it required invention to perceive that other members of the series could be used for the same purpose.

It is well understood by chemists that members of a homologous series will, in all probability, possess similar properties, although they may, of course, possess them in different degree. In fact, Dr. Smith (36) was quite positive that, on the basis of experiments with tricresyl phosphate, containing one carbon atom in the alkyl group, and tri-butyl phenyl phosphate, containing four carbon atoms in such group, he could predict the properties of the homolog of these compounds which contains two carbon atoms in the alkyl group. He was so certain of this that he considered further experiments unnecessary. This gives a clear indication of the recognized similarity between members of a homologous series. Dr. Lankelma (31), on cross examination, seemed to be less certain than Dr. Smith as to similarity between homologs, but it is thought to be clear that Dr. Smith's view is the correct one. Thus, in Webster's New International Dictionary, it is said that homology "is usually attended by a regular variation in physical properties". In Wertheim's Textbook of Organic Chemistry, published by The Blakiston Co. of Philadelphia, Copyright 1939, page 32, it is said that:

> The members are called homologs of one another. All of the aliphatic compounds belong to such series, and this fact greatly simplifies

> the study of organic chemistry. Instead of taking up one by one the thousands of known compounds, we are enabled to consider all of the compounds of any one homologous series at one time. These compounds have similar chemical traits because their structures are closely related; therefore we can learn the chemistry of the entire group with no more efforts than would otherwise be required to study a single compound. Of course we may anticipate certain "exceptions" to this general rule, but such exceptions will make very little trouble.

Similarly, in Chamberlain's Organic Chemistry, P. Blakiston's Sons, Philadelphia, 1934, Third Edition, page 21, a homologous series is defined as "A series of compounds the members of which differ in composition by a constant amount and whose physical constants change uniformly."

Under these circumstances it is thought to be clear that, when one member of a homologous series is found to be useful for a certain purpose, the idea of using the others for the same purpose does not involve invention, but merely routine experimentation. It is significant that the appellant's application recognizes the close similarity between the member of the present homologous series which includes one carbon atom in the alkyl group and those which include more. Thus, on page 3 of the application it is said:

> Broadly stated, this invention comprises a lubricating composition and method of lubricating under conditions of extreme pressure, wherein an addition agent comprised of an alkylated aromatic ester of an acid of phosphor-

> ous is employed in conjunction with a mineral oil base.

No distinction is made here as to the number of carbon atoms in the alkyl group and on page 4 of the application tri-cresyl phosphite, which contains only one carbon atom in this group is listed among the "examples illustrating but not intended to limit the type of addition agent contemplated by this invention." It seems apparent, therefore, that the applicant originally accepted as correct the position now taken by the Patent Office, namely, that the use of all members of the homologous series constituted a single broad invention. It was only when the lowest member of the series was found to be old that the theory that there was an inventive difference between this member and the others was advanced. Prior to this time the appellant, as shown for example by his original claim 3, now cancelled (43), was willing to claim the entire series as a single invention.

A situation very similar to the present one was considered by the United States Court of Customs and Patent Appeals in the case of *In re Hass et al,* 141 F. 2d 122. In that case, as in the present one, the applicant, after discovering that the member of a homologous series, having one carbon atom in the alkyl group was old sought to obtain a claim including all members of the series having two or more carbon atoms in this group. In holding this claim unpatentable, the Court said:

> It is well understood by chemists that members of a homologous series of chemical compounds possess the same principal character-

> istics; that generally the chemical and physical properties of the individual members vary gradually from member to member; and that *knowledge of the properties and chemical behavior of one of the members of the series suggests to the chemist the properties and behavior of the other members of the series.*" [Italics added.]

It is urged by the appellant that the tests described by Dr. Smith show a marked superiority of the compounds claimed here over the tricresyl phosphate of Downing. These tests, as the examiner points out in his report of February 7, 1945 (56–58), are confined to tributyl phenyl phosphate which has four carbon atoms in the alkyl group. No showing is made as to compounds having two carbon atoms in this group, although most of the claims include such compounds. Even after this was pointed out by the examiner, the appellant did not make such tests, but, on the basis of an alleged showing of superiority in the four carbon atom compounds, asks the Court to grant him a monopoly on all compounds of two or more carbon atoms. Manifestly there is no showing here which would warrant protection of such scope.

Further, even if it could be assumed that the compounds claimed were, as a group, unexpectedly superior to their homolog which is disclosed by Downing, this would not establish the presence of invention. Downing's disclosure of tricresyl phosphate amounts, to the skilled chemist, to a suggestion that its homologs, including tri-butyl phenyl phosphate, be tried. This seems to have been conceded by Dr. Smith (37).

Since it is obvious to try them, the mere fact, if such it be, that they are better than some chemists might have expected, cannot retroactively make the idea of using them patentable. It is in the nature of routine experiments that they sometimes produce unexpectedly good results. If results could always be accurately foreseen, no experiments would be necessary. It has been repeatedly held that the results of experimentation which was suggested by the prior art are not patentable no matter how useful they may be (*Naamlooze Venootschafs* v. *Coe,* 76 U. S. App. D. C. 313, 132 F. 2d 573; *In re Gauerke,* 86 F. 2d 330; *Minnesota Mining and Mfg. Co.* v. *Coe,* 79 App. D. C. 217, 99 F. 2d 986). As was said in the case last cited:

> But a showing of great industry in experimental research is not in itself sufficient to constitute invention, when the product thereof differs from those of the prior art only in degree and the result—no matter how useful it may be—is merely one step forward in a gradual process of experimentation.

For the reasons given it is submitted that, in view of Downing's disclosure of tricresyl phosphate, the use of the homologs of this substance which are disclosed and claimed by the appellant involved nothing more than routine experimentation and was, therefore, unpatentable.

CONCLUSION

It is submitted that the claims here involved recite nothing more than the use of homologs of tri-cresyl phosphate for a purpose to which that substance has already been put; and that such use would be obvious in view of the well-recognized relationship between homologs. It is further submitted that the claims are anticipated in all material respects by Downing's diloral cresyl phosphate. It is therefore thought that the decision appealed from should be affirmed.

Respectfully submitted.

W. W. COCHRAN,
Solicitor, U. S. Patent Office,
Attorney for Appellee.

E. L. REYNOLDS,
Of Counsel.

OCTOBER 1947.

U. S. GOVERNMENT PRINTING OFFICE: 1947